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SNAP 11 SURVEYOR PROGRAM  
THIRTEENTH QUARTERLY PROGRESS REPORT

[U]

MND-2952-30

February 1, 1965 Through April 30, 1965  
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FOREWORD

This thirteenth quarterly progress report describes the activities on the SNAP 11 program (Surveyor). It covers work performed from February 1, 1965 through April 30, 1965, under Contract AT(30-1)-2952 with the U.S. Atomic Energy Commission.

Previous quarterly reports issued under the SNAP 11 Surveyor Program are:

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|--------------|--|
| MND-P-2811-1 | SNAP 11, Surveyor Program<br>Quarterly Progress Report<br>No. 1, February 1 through<br>April 30, 1962.         |
| MND-P-2811-2 | SNAP 11, Surveyor Program<br>Quarterly Progress Report<br>No. 2, May 1 through July<br>31, 1962.               |
| MND-P-2811-3 | SNAP 11, Surveyor Program<br>Quarterly Progress Report<br>No. 3, August 1 through<br>October 31, 1962.         |
| MND-P-2811-4 | SNAP 11, Surveyor Program<br>Quarterly Progress Report<br>No. 4, November 1, 1962<br>through January 31, 1963. |
| MND-P-2811-5 | SNAP 11, Surveyor Program<br>Fifth Quarterly Progress Re-<br>port, February 1 through April<br>30, 1963.       |
| MND-P-2811-6 | SNAP 11, Surveyor Program<br>Sixth Quarterly Progress Re-<br>port, May 1 through July 31<br>1963.              |
| MND-P-2811-7 | SNAP 11, Surveyor Program<br>Seventh Quarterly Progress<br>Report, August 1 through<br>October 31, 1963.       |

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MND-2952-8	SNAP 11, Surveyor Program Eighth Quarterly Progress Re- port, November 1, 1963 through January 31, 1964.
MND-2952-10	SNAP 11, Surveyor Program Ninth Quarterly Progress Re- port, February 1 through April 30, 1964.
MND-2952-14	SNAP 11, Surveyor Program Tenth Quarterly Progress Re- port, May 1 through July 31, 1964.
MND-2952-20	SNAP 11, Surveyor Program Eleventh Quarterly Progress Report, August 1 through October 31, 1964.
MND-2952-26	SNAP 11, Surveyor Program Twelfth Quarterly Progress Report, November 1, 1964 through January 31, 1965.

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### SUMMARY

Dynamic tests at flight acceptance and qualification levels were completed on an operating thermal control system employing the S/N 1 generator as the thermal test vehicle. Results indicated that system calibration could be retained at qualification shock, vibration, and acceleration levels with the shutter door latched in the open position, although minor damage was evidenced by slight shredding of door insulation, rotation of door on mounting shaft, and loosened locking nuts. Continued testing at flight acceptance levels with the door unlatched and allowed to float free proceeded without incident; however, further testing at qualification vibration levels showed a gradual increase in friction within the door shaft bearings followed by rupture of a liquid metal tube within the redundant NaK hydraulic system. Rupture of the tubing occurred on the 69th vibration sweep and was believed due to loss of a tube support clip and possibly metal fatigue in the heat-affected area adjacent to a weld.

Thermal coupling investigations were performed on the S/N 1 generator at lunar day thermal vacuum conditions. The addition of reflective thermal insulation consisting of fired-gold coatings on stainless steel foil in the generator inner cavity, adjacent to the NaK reservoirs and inner heat shields, proved effective. Shutter door angular openings of 35 degrees, corresponding to an increase in NaK system temperature of 30° F, were obtained during an average hot junction temperature increase of 64° F.

Life testing of the S/N 4 modified generator was completed on March 9, 1965 after more than 3000 hours at operating temperature. Internal electrical resistance at temperature increased from 0.339 to 0.384 ohm. Power output, normalized to hot and cold design temperatures of 925° and 350° F, respectively, decreased from 26.34 to 23.16 watts. Some of the 12.1% decrease in power output can be attributed to thermal cycles during the extensive test program.

Upon completion of S/N 4 modified generator life testing, a replacement thermal control system, previously NaK-filled and bench tested at operating temperature, was installed. In addition, the gold-plated emissive coatings on the heat shields and shutter door assembly were replaced with fired-gold coatings of the type tested in the S/N 1 generator. Thermal vacuum evaluations on the NaK thermal control system--hot junction thermal coupling effects were under way at the end of this report period. Data showed that the addition of nondeteriorating emissive foils in the generator cavity caused the thermal control system to become more responsive to changes in hot junction temperatures in a vacuum environment. Further work will be performed on thermal coupling and the results will be applied to the Q series generators for improved thermal performance.

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Component development was continued during this report period. In addition to the fired porcelain and gold emissive coatings evaluated on the S/N 1 and S/N 4 modified generators, effort was expended on testing mica electrical insulation and lubricants, and determining impurities within the generator that could be potential causes of power degradation.

Fabrication of the remaining components for the Q/N 1 and Q/N 2 generators, suspended in November 1964, was resumed. Assembly of the Q/N 1 unit was initiated. Results of the recent thermal control system investigations and component development, combined with revised assembly procedures to utilize dry box assembly, are being employed on the Q series.

Fuel interface coordination activities were resumed with Oak Ridge National Laboratories. Current plans are to fuel the Q/N 1 generator with curium-242 at ORNL in August 1965 and perform a 120-day demonstration test. As a result, efforts to support the fueling were initiated late in this report period. Activities currently under way include providing fuel block assemblies, a safety analysis hazards report, and fuel handling tools to accommodate the fueling operation.

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## I. INTRODUCTION

During the first and second quarters (February through July 1962) of the SNAP 11 Development Program, the Martin Company devoted time to interface coordination and program definition with the Surveyor spacecraft contractor, Hughes Aircraft Company. Also during this period, the generator configuration was defined to produce 25 watts (e) under lunar night conditions for a period of 130 days following fueling. Oak Ridge National Laboratory (ORNL) was selected by the U. S. Atomic Energy Commission to process and encapsulate the curium-242 for two radioisotope thermoelectric generators (RTG). Regulated power will be provided at 29 volts through an 85% efficient, dc-to-dc converter to complete the power package.

The third and fourth quarters (August 1962 through January 1963) were utilized for detail analysis and design of the generator. Fabrication of thermoelectric modules, the NaK thermal control system, and a thermal mockup of the generator was completed. Dc-to-dc converter breadboard, module, and thermal control system tests were performed. The safety criteria for fuel containment were clarified, specifying intact atmospheric re-entry; and program for fuel block high velocity impact and simulated atmospheric tests was begun.

The fifth and sixth quarters (February through July 1963) were devoted to fabrication and testing of two prototype generators, S/N 1 and S/N 3. Tests on the generators included thermal environments of lunar night and day conditions. The thermoelectric module life test program thermal mockup heat transfer tests, and thermal control system tests were completed. Half-scale, high velocity impact tests were performed. Engineering was initiated to fabricate two additional prototype generators for system integration tests by Hughes. The detail design of prototype dc-to-dc converters to step up and regulate generator output power was started in July 1963.

The seventh and eighth quarters (August 1963 through January 1964) brought about the completion of the 120-day life test and post-test examination of the first prototype unit, S/N 3. Performance tests, including qualification level vibration, acceleration, and shock, were completed on Prototype Unit S/N 1. Radiation dose rates around the curium-242 generator were calculated, and the detail design of a shipping cask for the fuel block assembly was completed. Four packaged dc-to-dc converters were fabricated and performance evaluated. Engineering design studies were completed and detail fabrication of Prototype Generators S/N 2 and S/N 4 initiated.

During the ninth and tenth quarters (February through July 1964) the S/N 2 and S/N 4 generators were assembled and performance

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evaluated at room temperature and simulated lunar conditions. Environmental dynamic tests were completed on the converters and a generator-converter system was supplied to Jet Propulsion Laboratory (JPL) for further evaluations. The safety analysis and test efforts, based on existing safety criteria, were completed. Reliability engineering, design revisions and procurement of materials for four qualification generators were initiated.

The eleventh and twelfth quarters (November 1964 through January 1965) have been devoted to fabrication of details for four qualification generators, the assembly and extensive tests of the S/N 4 modified generator and conducting of development programs to improve existing components. These development programs covered areas such as thermal control system tests, emissive coatings, and electrical insulation. The alternate fuel analysis using Po-210 and Pm-147 was completed. Reliability engineering continued but the safety and d-c converter areas were inactive.

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This past quarter was devoted primarily to thermal control system investigations and component improvement. Dynamic testing of the S/N 1 generator containing an operating control system was performed at both flight acceptance and qualification levels. A 3000-hour life test was completed on the S/N 4 modified generator, a replacement thermal control system was installed, and additional thermal vacuum testing was accomplished. Assembly of the Q/N 1 and Q/N 2 generators was started. A safety hazards report and fuel block fabrication, in support of Q/N 1 generator fueling at ORNL, was also initiated.

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## II. GENERATOR DEVELOPMENT PROGRAM\*

### A. OBJECTIVES

The objectives of the thermoelectric generator development program for the past quarter were to:

- (1) Complete the 120-day life test of S/N 4 modified generator.
- (2) Develop and improve thermal control system response and thermal coupling with generator hot junction temperatures at vacuum test conditions.
- (3) Evaluate the S/N 1 generator mockup with a thermal control system under flight acceptance and qualification level dynamic test conditions.
- (4) Demonstrate ability to calibrate the thermal control system at desired hot junction temperatures after installation in a vacuum environment at lunar night temperatures.
- (5) Incorporate design modifications into the S/N 4 modified generator and subject it to flight acceptance and qualification, thermal vacuum and dynamic tests.
- (6) Continue the component development program on low emissivity gold coatings and on natural mica for use as an electrical insulator, evaluate Molykote X-15 as a lubricant, and fill a third thermal control system with NaK and check out prior to installation in a generator.
- (7) Initiate fabrication of the Q/N 1 and Q/N 2 generators.

### B. SCOPE

#### 1. S/N 4 Modified Generator Tests

Evaluations of the S/N 4 modified generator were begun late in the eleventh quarterly reporting period. This rebuilt generator was used to demonstrate a design objective of 25 watts (e) for a 600-watt (t) input under lunar night conditions and to gather basic parametric data on generator performance, including the heat dumping ability of the thermal control system.

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\*W. Brittain, D. Ganz, E. Courtney, W. McNerney and G. Numsen

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Fig. 1. Generator Test Console

The major changes made in modifying the original S/N 4 prototype generator, prior to this test series include:

- (1) Increased fin length
- (2) Redesigned shutter door
- (3) Redesigned shutter door actuator links
- (4) Added adjustable spring tension mechanism to shutter door
- (5) Redesigned inner heat shield
- (6) Replaced thermoelectric modules
- (7) Added adjustable bellows to piston bellows.

a. Test facilities

The S/N 4 modified generator while on life test was operated from a generator test console similar to the one shown in Fig. 1. This console contains a variable resistive load to match the generator output, regulated a-c power, a temperature controller and monitoring positions for temperature, load voltage, load current, and power input.

Facilities for pressurizing the generator with cold trapped argon and for evacuating the generator are located behind the test console. For the purpose of evacuating the generator, a Welsh Model 1397 Duo-Seal vacuum pump is used and is capable of a pumping speed of 425 liters per minute.

Temperature. The S/N 4 modified generator was instrumented with 74 chromel-alumel thermocouples. All thermocouples, excepting Nos. 2, 5, and 8 (middle hot junction, middle cold junction and middle heater block, respectively) were wired to a stepping switch to reduce the number of electrical connections to be made whenever the generator is relocated. The thermocouples were read with a Rubicon Model 2745 potentiometer giving an overall system accuracy of about 1%.

Electrical power. Power input to the generator was measured with a Weston Model 310 wattmeter. Power input accuracy was  $\pm 0.25\%$  of full scale.

Output power. The output power was determined from the internal voltage across the elements and from the load current. A 50-amp 50-mv Weston shunt was used with a Leeds and Northrup Model 8667 millivolt potentiometer to read the load current. The load voltage was measured with an Electro Instruments Model 8409 MB digital voltmeter. Output power accuracies are  $\pm 0.5\%$ .

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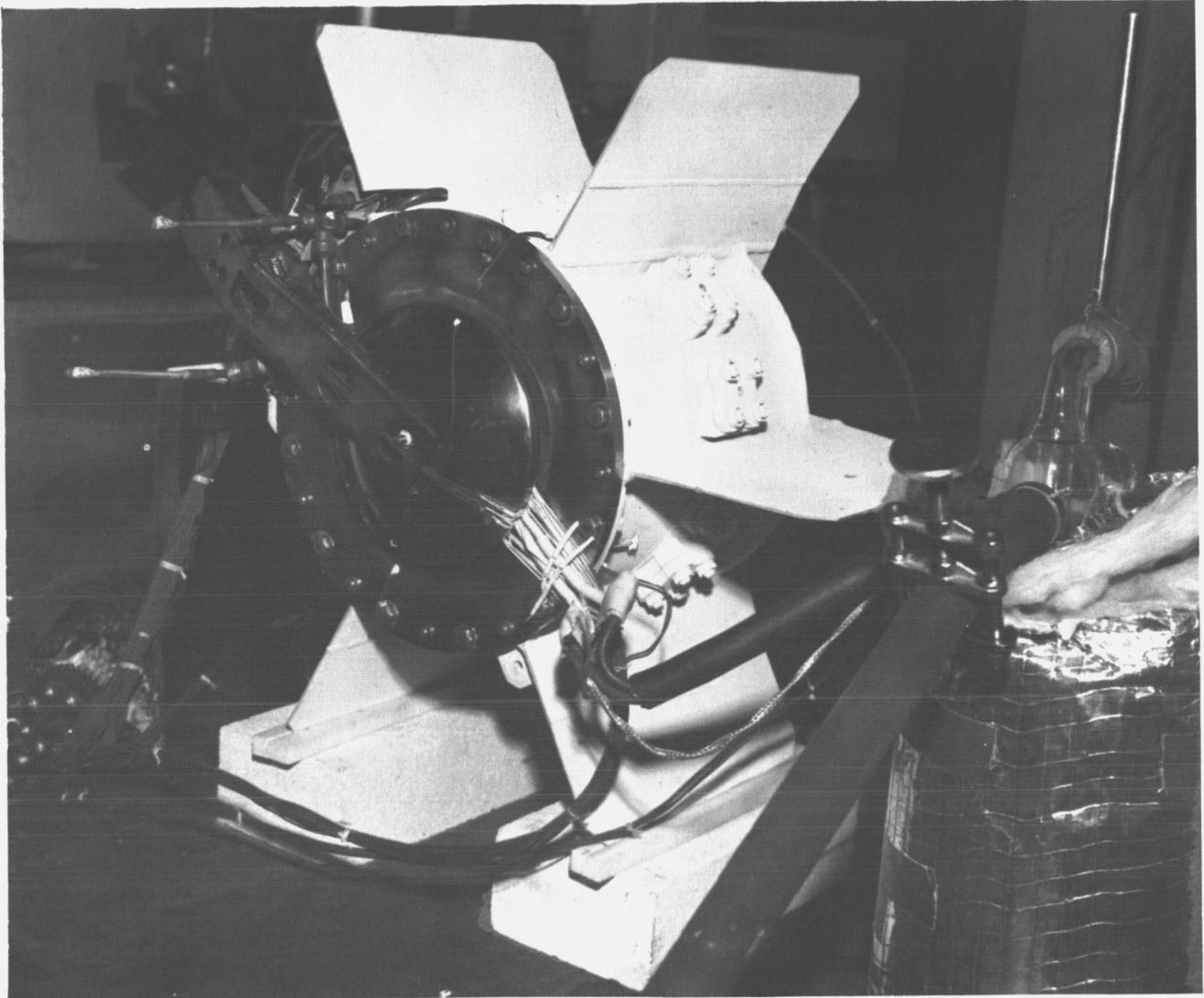


Fig. 2. S/N 4 Modified Generator Life Test

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Generator internal pressure. The internal pressure of the generator was measured by means of a mercury U-tube manometer and by a NRC thermocouple-type vacuum gage.

b. Life test

At the start of this reporting period the S/N 4 modified generator has been on life test for approximately 90 days. For this test the position of the generator has been as shown in Fig. 2, in air, with the instrumented thermoelectric module located on the lower side of the generator.

During the latter part of the life test the insulation resistance between the thermoelectric elements and the generator case was obtained. This was in addition to the other basic thermal and electrical data taken. The life test of the generator was concluded on March 9, 1965 after approximately 127 days of operation at elevated temperatures and the generator transported to Manufacturing to incorporate the following:

- (1) Installation of a thermal control system which had been filled with NaK, checked out at operating temperature in a mockup, coarse calibrated and the crimping operation completed.
- (2) Replacement of the discolored gold plated emissive coatings on the inner heat shields and the shutter door components with the recently developed ceramic fired Solaramic enamel barrier in conjunction with Hanovia Liquid Bright Gold.
- (3) Installation of gold coated stainless steel foil between the NaK ring and generator inner cavity to improve the thermal coupling with the hot junction.

A tabulation of normalized power output, generator internal resistance and days at elevated temperature obtained in the life test of the S/N 4 modified generator is contained in Table 1 and plotted in Fig. 3. Plots of average hot junction temperature, average cold junction temperature, power output, internal resistance, and  $E_{oc}/T$  (approximation of Seebeck coefficient) versus time are shown in Fig. 4. The upward trend in internal resistance can be accounted for in part by the three thermal cycles the generator experienced. The decrease in power output has been attributed to the increase in internal resistance. A correlation between the change in internal resistance and the change in power output is shown in Fig. 5. The rate of power decrease during the final period of constant temperature operation covering 1320 hours (January 14 to March 9) tended to decrease and averaged 3.6% per 1000 hours.

TABLE 1  
Selected Test Data--S/N 4 Modified Generator

<u>Date</u>	<u>Power Output,* Normalized (watts)</u>	<u>Internal Resistance (ohm)</u>	<u>Days at Elevated Temperature</u>
10/27	26.34	0.339	2
10/28	26.31	0.337	3
11/3	26.85	0.337	9
11/4	26.96	0.337	10
11/6	26.73	0.336	12

Generator shutdown (planned) for transfer from Environmental Facility to Small Power Plant Laboratory.

12/1	26.24	0.342	29
12/4	25.70	0.344	32
12/14	25.57	0.344	42
12/16	25.20	0.350	44

Power outage to generator heaters (unplanned)

1/7	25.09	0.360	65
1/11	24.13	0.362	69

Partial generator shutdown (planned). Decision made to continue life tests.

1/14	24.35	0.363	72
1/18	24.64	0.366	76
1/22	24.33	0.367	80
1/25	24.06	0.370	83
2/1	24.43	0.377	91
2/8	23.89	0.372	98
2/10	23.59	0.374	100
2/15	23.71	0.370	105
2/22	23.33	0.375	112
2/23	23.65	0.373	113
2/25	23.40	0.381	115
3/1	23.00	0.378	119
3/4	23.27	0.383	122
3/5	23.17	0.383	123
3/8	23.17	0.384	126
3/9	23.16	0.384	127

\*Normalized to design junction temperatures of 925° F hot junction and 350° F cold junction.

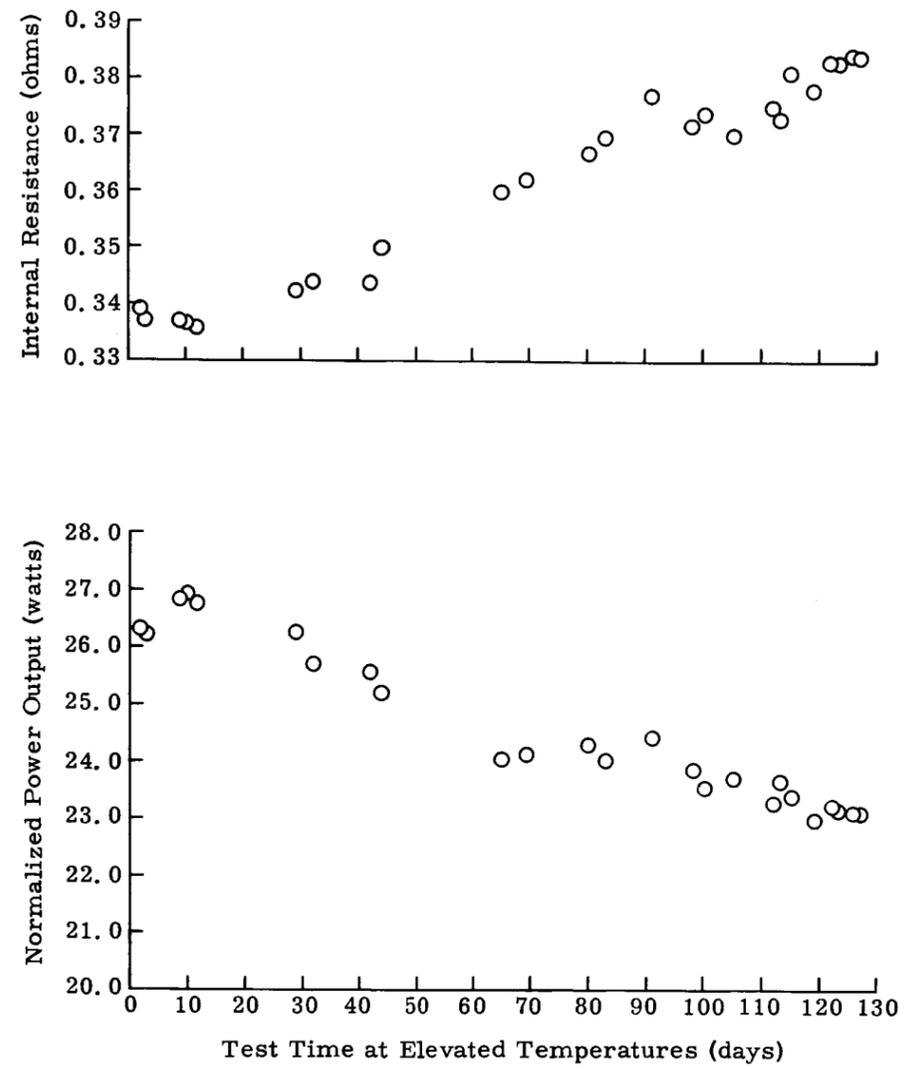


Fig. 3. SNAP 11 S/N 4 Modified Generator Normalized Power Output and Internal Resistance Versus Test Time

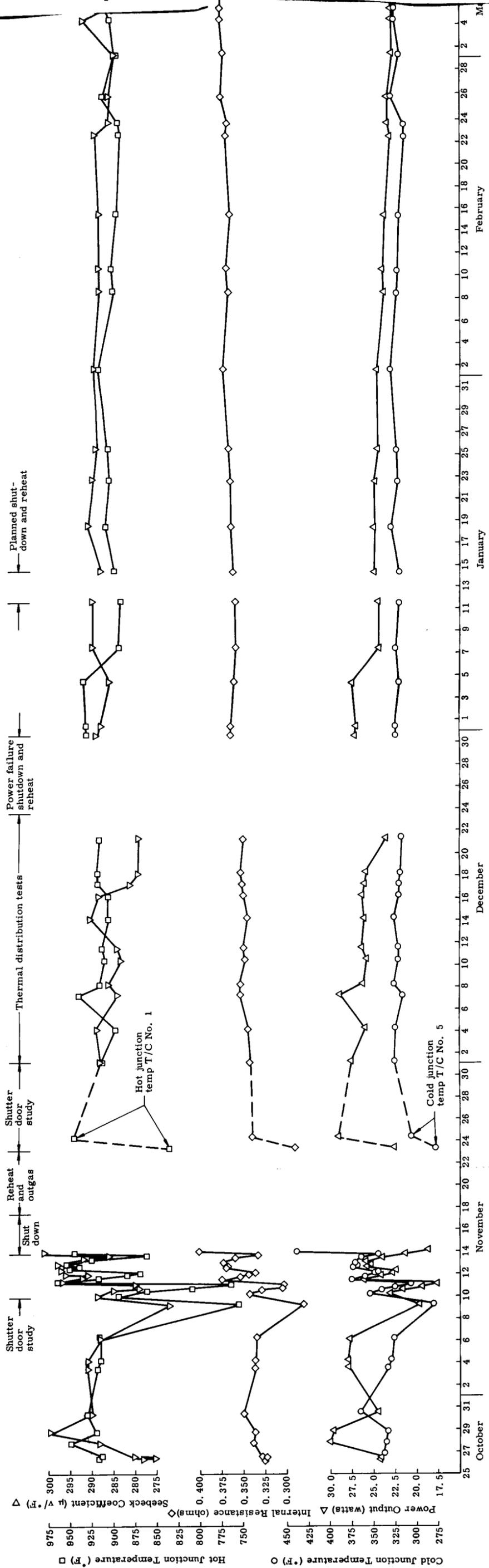


Fig. 4. S/N 4 Modified Generator Performance Character

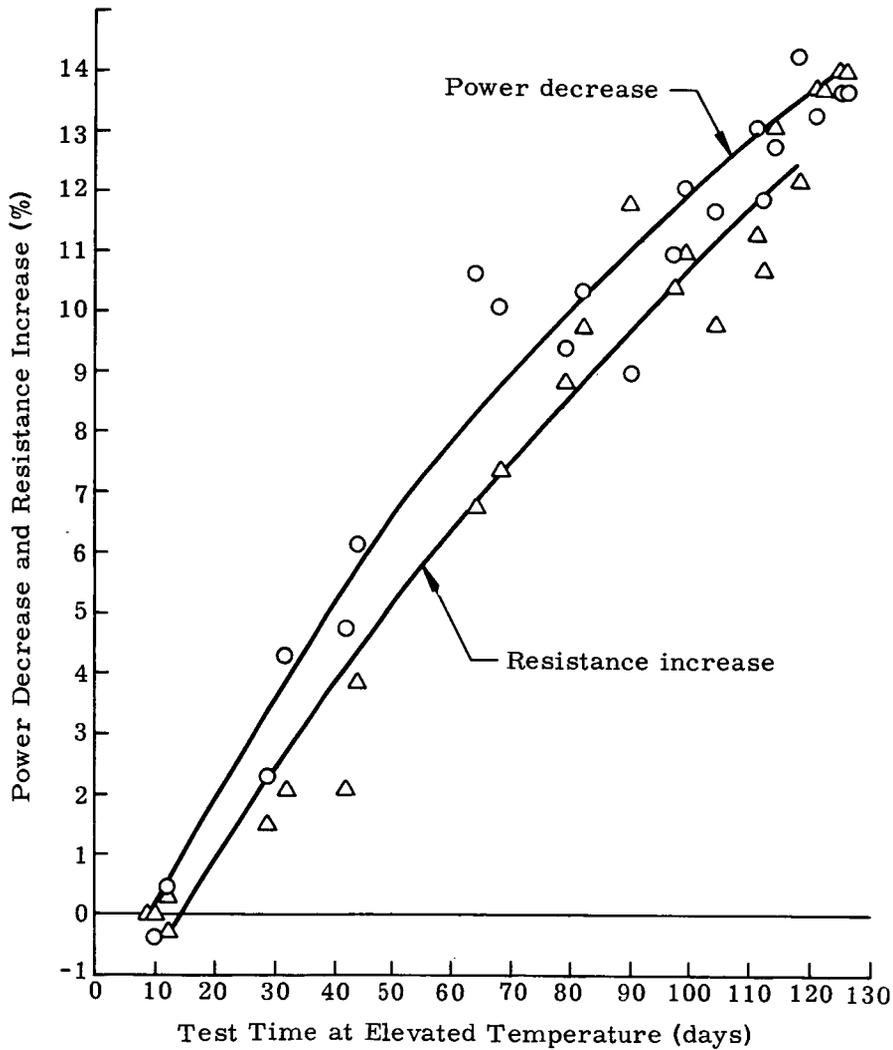


Fig. 5. SNAP 11 S/N 4 Modified Generator Power Output Decrease and Internal Resistance Increase Versus Test Time (derived from data of Fig. 4)

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During the life test, measurements were made to determine the electrical insulation resistance between the thermoelectric circuit and the generator outer housing. The resistance was determined by measuring the voltage existing between the generator output terminals and the generator case and the current by shorting the output terminal to the case using a low impedance shorting wire and a current shunt. This measurement was made first from one side of the generator output and then from the other. The two values of resistance that were obtained differed at most by approximately one ohm and ranged between 21 and 36 ohms. Calculations indicate that the total power loss due to leakage was less than 0.5% of the total generator electrical output.

## 2. S/N 1 Generator Mockup Thermal Coupling Tests

A new thermal control system, filled with NaK and operated at temperature on the bench, was installed into the S/N 1 prototype generator. This unit was intended as a dynamic test bed to determine the ability of the shutter mechanism to remain in calibration when subjected to dynamic loads imposed by flight acceptance and qualification level dynamic tests. However, the mockup was initially subjected to thermal testing under lunar day vacuum conditions in an effort to:

- (1) Obtain additional thermal data on the relationships (thermal coupling) between the thermoelectric hot junction and NaK ring temperatures during shutter door operation, and the heat dump capability of an operating shutter door.
- (2) Evaluate the mechanical operation of the shutter door.

The generator mockup consisted of the previously dynamic tested S/N 1 prototype generator with a NaK-filled thermal control system (TCS 1) replacing the Sanowax-filled unit originally installed in the generator. In addition, the gold-plated surfaces of the shutter door were recoated, a new inner heat shield was installed, and the aluminum oxide and chromium oxide emissive coatings on the surfaces on the heater block were renewed. The thermoelectric performance of this earlier prototype generator was poor, due to previous test operations, and therefore, thermal coupling data has to be evaluated carefully to avoid misinterpretation.

At no time during these tests was any attempt made to obtain data on thermoelectric operation; at the initiation of thermal testing, the thermoelectric output leads were shorted and remained so throughout the evaluation. The thermal data obtained from this test series cannot be interpreted as being absolute, since the basic generator employed did not duplicate the present design configuration, being an earlier model which had accumulated considerable test time prior to these tests. However, the trends indicated are applicable in evaluating future generator design and test direction.

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The thermal testing of the generator mockup, in a lunar day vacuum environment, can be divided into three phases:

- (1) The generator (designated S/N 1\*) was first tested with the inner heat shield and shutter door gold surfaces plated by the process used on all previous generators (gold plated directly on the stainless steel surfaces without any intermediate barrier coating).
- (2) An investigation of the gold plate deterioration problem was performed and a replacement coating process, using an intermediate ceramic layer between the gold and the surface being coated, was developed. It was used to coat the gold surfaces of the generator. This generator (designated S/N 1\*\*) was then thermally tested.
- (3) For the third phase of testing, 14 layers of gold-coated, one-mil stainless steel foil were installed around the inner periphery of the heat shield in the S/N 1\*\*. This generator (designated S/N 1\*\*\*) was then thermally tested.

a. S/N 1\* test

Result of the S/N 1\* testing are shown in Figs. 6 and 7. From Fig. 6, a change in the hot junction temperature of 162° F was required to open the shutter door 35 angular degrees, with a corresponding increase in the NaK ring temperature of 28° F.

From Fig. 7, the power dumped at a shutter door opening of 35 degrees was 320 watts (extrapolated).

Since the thermal control system is designed so that a 57° F change in the NaK ring temperature is required to cause the door to go from closed (0 degree) to fully open (70 degrees), it is seen that the shutter door was operating mechanically as designed. However, the thermal coupling between the hot junction and NaK ring temperatures was inadequate.

b. S/N 1\*\* test

After the completion of the S/N 1\* thermal and dynamic testing, the gold plating on the heat shield and shutter door surfaces had oxidized to the extent that the surfaces were essentially black. An investigation of this deterioration problem resulted in the application of a coating process which consisted of adding a ceramic layer between the gold

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\*Indicates modifications to generator

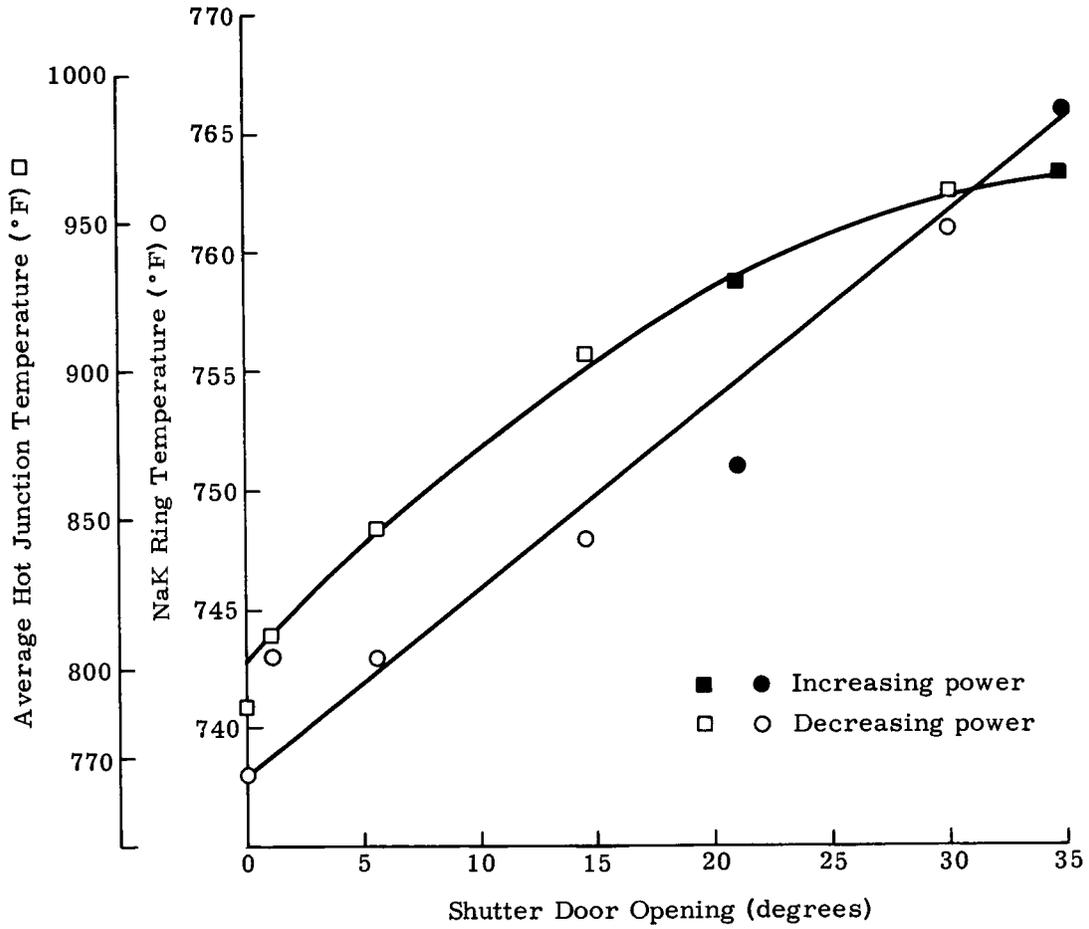


Fig. 6. S/N 1\* Temperature Distribution as a Function of Shutter Door Opening (lunar day conditions)

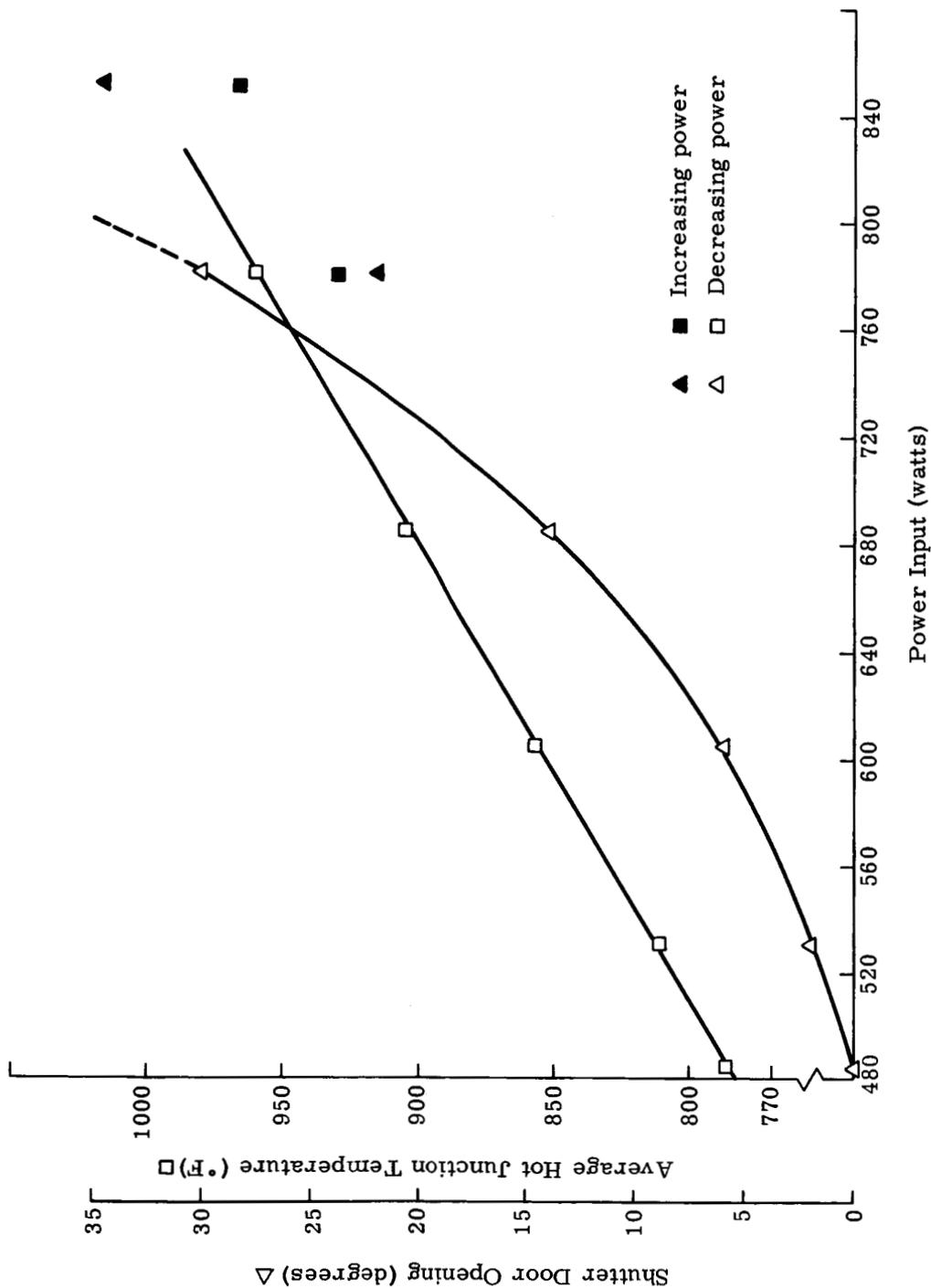


Fig. 7. S/N 1\* Characteristics as a Function of Power Input (lunar day conditions)

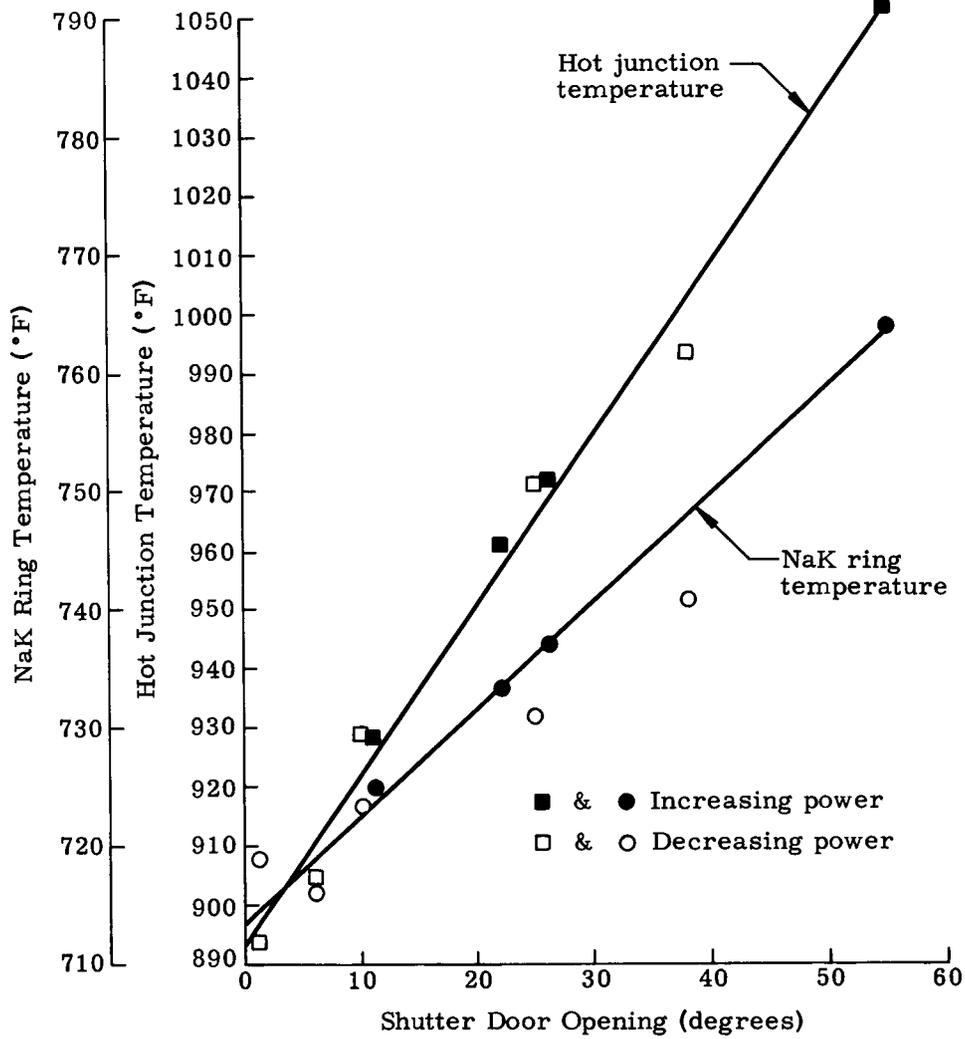


Fig. 8. S/N 1\*\* Temperature Distribution as a Function of Shutter Door Opening (lunar day conditions)

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coat and the stainless steel base metal. All gold surfaces of the shutter door and inner heat shield were then coated, using the new process.

After completion of the S/N 1\* qualification level dynamic testing, the thermal control system was shown to be out of calibration. Calibration was effected when the system yielded due to overpressuring during a temperature calibration cycle. Since the hot junction temperature required to operate the shutter door after system yielding was excessive, the connecting links between the actuator pistons and shutter door cams were altered to initiate earlier door opening. A 3/8-inch insert was welded into the slots of the two existing links to reduce the maximum hot junction temperature required to initiate door opening (in air) from 1054° to 1018° F.

It should also be pointed out that, during the qualification level vibration testing of the S/N 1\*, the diaphragm seal member adjacent to the NaK rings cracked, resulting in loss of the argon cover gas. Thus, in testing of the S/N 1\*\* (and S/N 1\*\*\*), a vacuum existed in the thermo-electric converter section during lunar day testing.

Results of testing at simulated lunar day conditions are shown in Figs. 8 and 9. From Fig. 8, the change in the average hot junction temperature required to cause a 35-degree door opening was 101° F and the corresponding change in the NaK ring temperature was 32° F.

Figure 9 is a plot of power input versus door opening. For a 35-degree shutter door opening, the power dumped is approximately 220 watts. (Some discrepancy in the power input was noted during this testing, and it is not certain where the power curve should lie in Fig. 9.)

c. S/N 1\*\*\* test

After completion of the S/N 1\*\* testing, gold-coated nickel foil was positioned around the inner periphery of the heat shield in an effort to improve the thermal coupling. Results of testing under simulated lunar day conditions were as shown in Figs. 10 and 11. As shown in Fig. 10, to open the shutter door 35 degrees, required a 64° F change in the average hot junction temperature and a corresponding 30° F change in the NaK ring temperature. Thus, the hot junction temperature change required was reduced from 101° F (S/N 1\*\* tests) to 64° F, a significant improvement in thermal coupling by placing gold-coated foil on the inner periphery of the heat shield.

From the data in Fig. 11, the heat dump capability of the shutter door when opened 35 degrees is approximately 320 watts.

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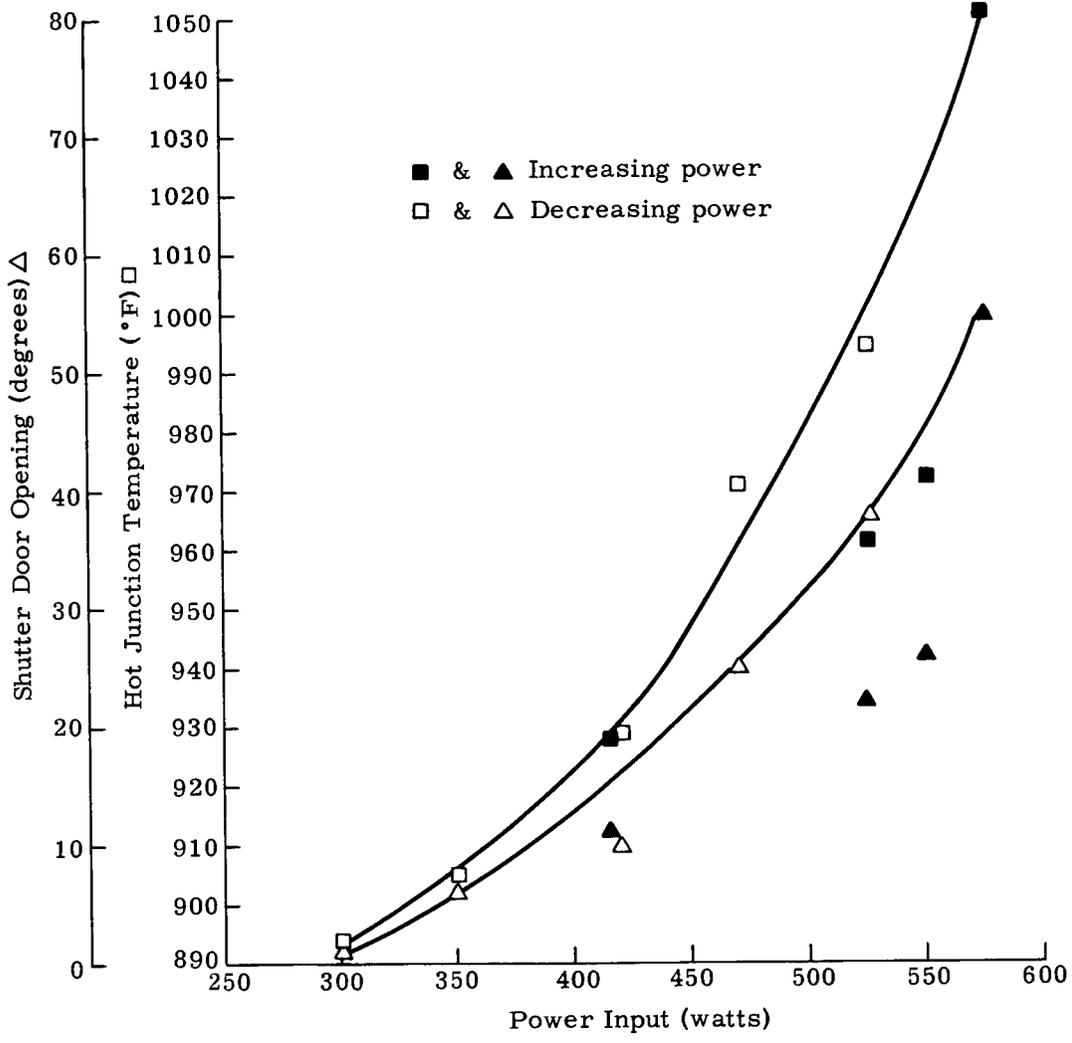


Fig. 9. S/N 1\*\* Characteristics as a Function of Input Power (lunar day conditions)

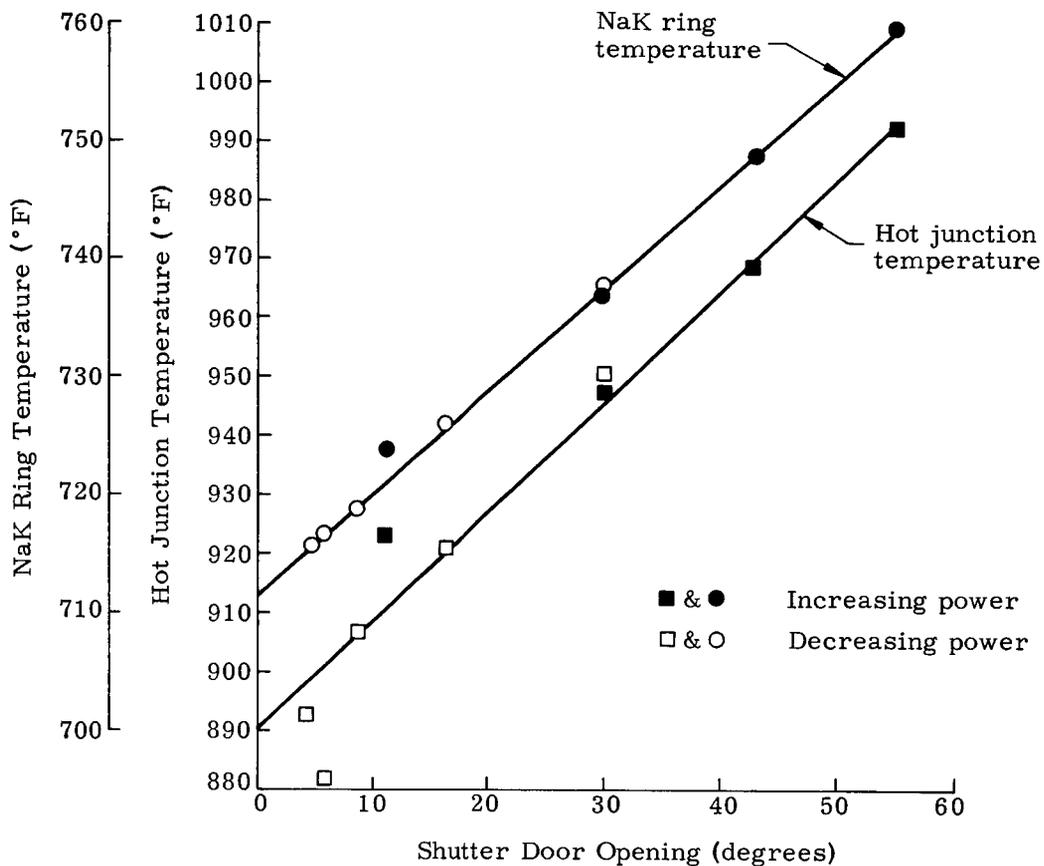


Fig. 10. S/N 1\*\*\* Temperature Distribution as a Function of Shutter Door Opening (at lunar day conditions)

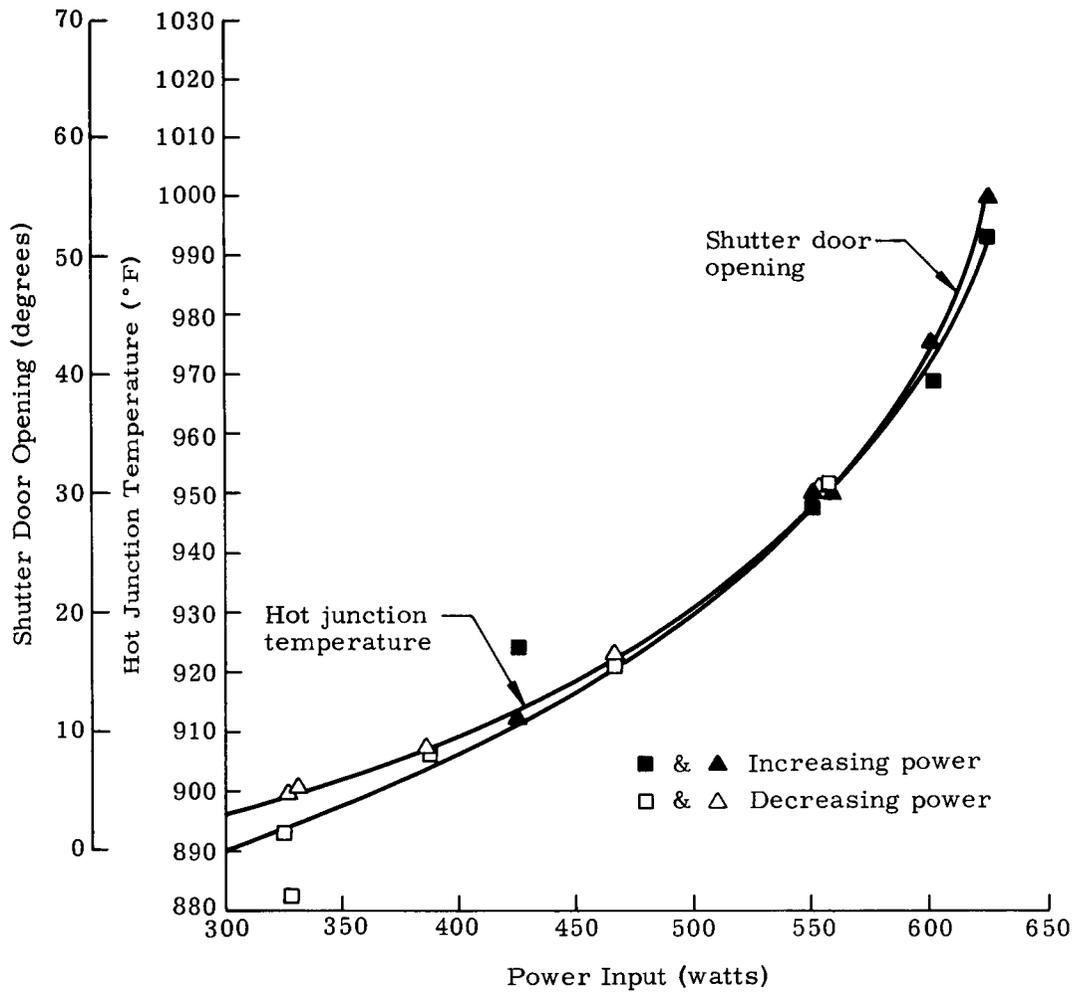


Fig. 11. S/N 1\*\*\* Characteristics as a Function of Input Power (lunar day conditions)

d. Conclusions

Table 2 and Fig. 12 compare the major results obtained from the S/N 1 (mockup generator) lunar day vacuum testing. It is readily apparent that the ceramic-fired gold coating, which exhibited excellent stability during 1000° F vacuum testing, and the gold-coated foil resulted in improved thermal coupling.

TABLE 2  
Comparison of Generator Mockup Tests (1)

<u>Generator Designation</u>	<u>Δt Hot Junction (° F)</u>	<u>Δt NaK Ring (° F)</u>	<u>Power Dumped (watts)</u>
S/N 1*	162	28	320
S/N 1**	101	32	220
S/N 1***	64	30	320

(1) All values are for 0 to 35 degrees of door opening.

3. S/N 1 Generator Mockup Dynamic Tests

After completing the thermal test of the S/N 1\*, the generator mock-up was subjected to dynamic testing of the flight acceptance and qualification levels required in Martin Specification MN 10117. The purpose of these tests was to verify system design and to determine the ability of the shutter mechanism to remain in calibration at operating temperature during critical dynamic launch environments. The qualification tests are the most rigid and are intended to simulate the effects of the Atlas-Centaur and of retro-engine burning during launch boost and descent to the lunar surface. The flight acceptance tests are designed to ensure flight quality assemblies with a minimum effect on endurance limit and functional tolerances.

a. Dynamic test levels

Vibration test. The generator is vibrated in a direction parallel to the thrust axis and in two critical orthogonal directions perpendicular to the thrust axis.

The generator is subjected to a sinewave sweep from 5 to 26 cps at 0.50-inch double amplitude and 26 to 1500 cps at ±18 g. The sinewave sweep is logarithmic from 5 to 1500 cps over a period of two minutes. A random noise of 4.5 g, rms band limited from 100 to 1500 cps, is superimposed on the sinusoidal vibration. The combined sinusoidal and

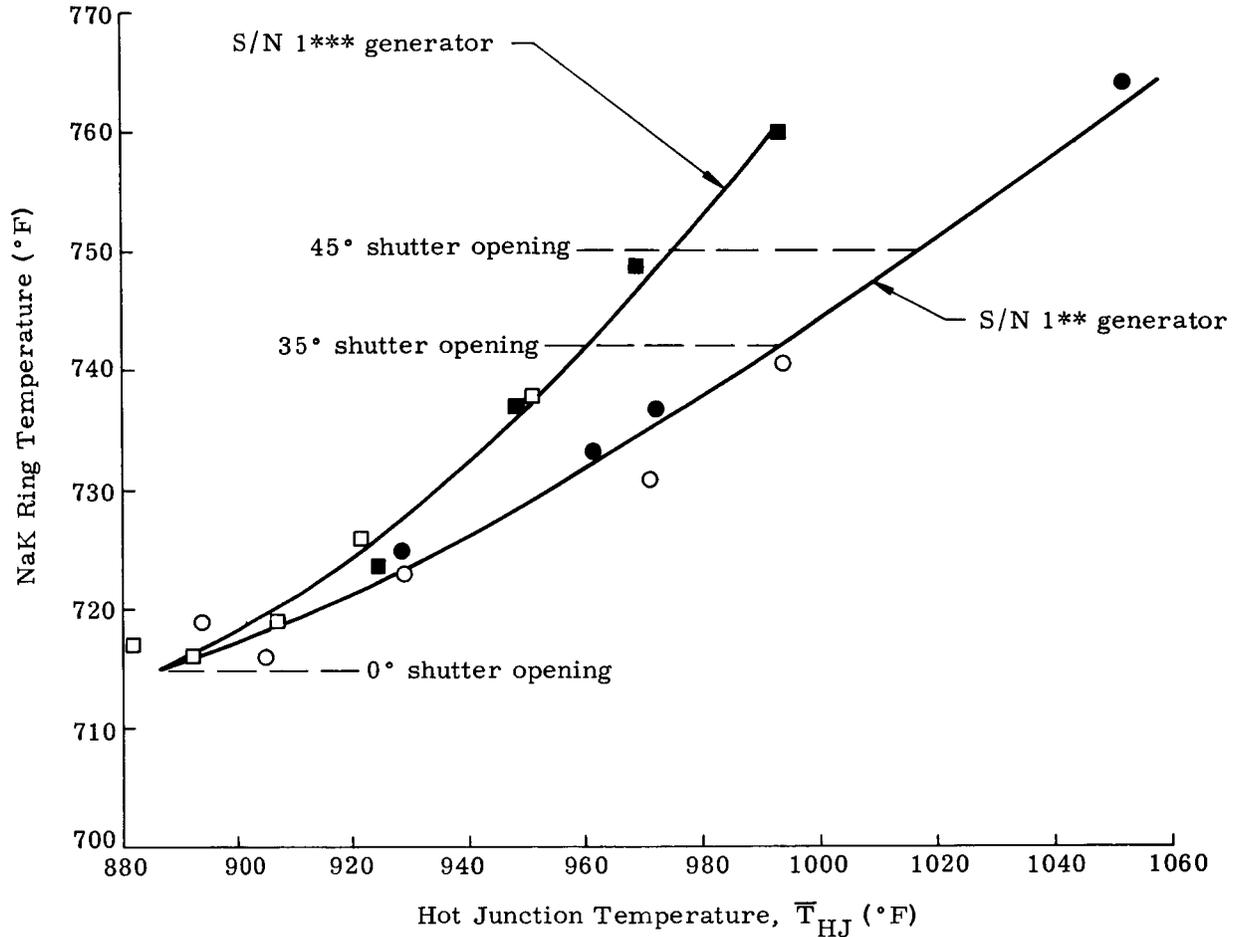


Fig. 12. Comparison of S/N 1\*\* and S/N 1\*\*\* Thermal Coupling (lunar day conditions)

random noise loads are repeated five more times for a total of 6 cycles, except that the random noise level is reduced to 2 g rms for the additional five cycles.

Acceleration test. The maximum steady-state acceleration is applied parallel to the thrust axis. The test levels are 10 g for 4 minutes and 5 g for 7 minutes.

Shock test. Shock tests simulate the environments caused by the retro-engine and touchdown dynamics and encountered by the spacecraft vehicle during the descent phase. The generator is subjected to four 5-msec, 25-g half-sinewave shocks along the positive thrust axis and to four (2 in each direction) 5-msec, 15-g half-sinewave shocks along each of two orthogonal axes perpendicular to the thrust axis.

Acceleration test. The generator is subjected to a 6 g steady-state acceleration of 5 minutes duration along the spacecraft thrust axis.

Vibration test. The vibration loads are applied parallel to the thrust axis and also in each of two orthogonal axes perpendicular to the thrust axis. The total test time in each orthogonal direction is 6 minutes. The test is as follows:

- (1) 5 to 20 cps at 0.3 inch double amplitude displacement;
- (2) 20 to 150 cps at 6 g peak;
- (3) 150 to 1500 cps at 2 g peak.

The sinusoidal sweep is logarithmic at such a rate that the frequency range from 5 to 1500 cps consumes 2 minutes. It is conducted three times for a total test of six minutes along each of the three axes.

b. Test conditions

The vibration testing was performed in three phases:

- (1) The shutter door bracket was first rigidly attached to the generator outer housing in the fully open position and the generator subjected to the flight acceptance level acceleration and vibration tests, and the qualification level acceleration, shock and vibration tests.
- (2) The door bracket was then unbolted from the housing, thus allowing free shutter motion, and the generator again subjected to the flight acceptance level acceleration and vibration tests. The generator was then subjected to the qualification level vibration test in the vertical (thrust axis) only.

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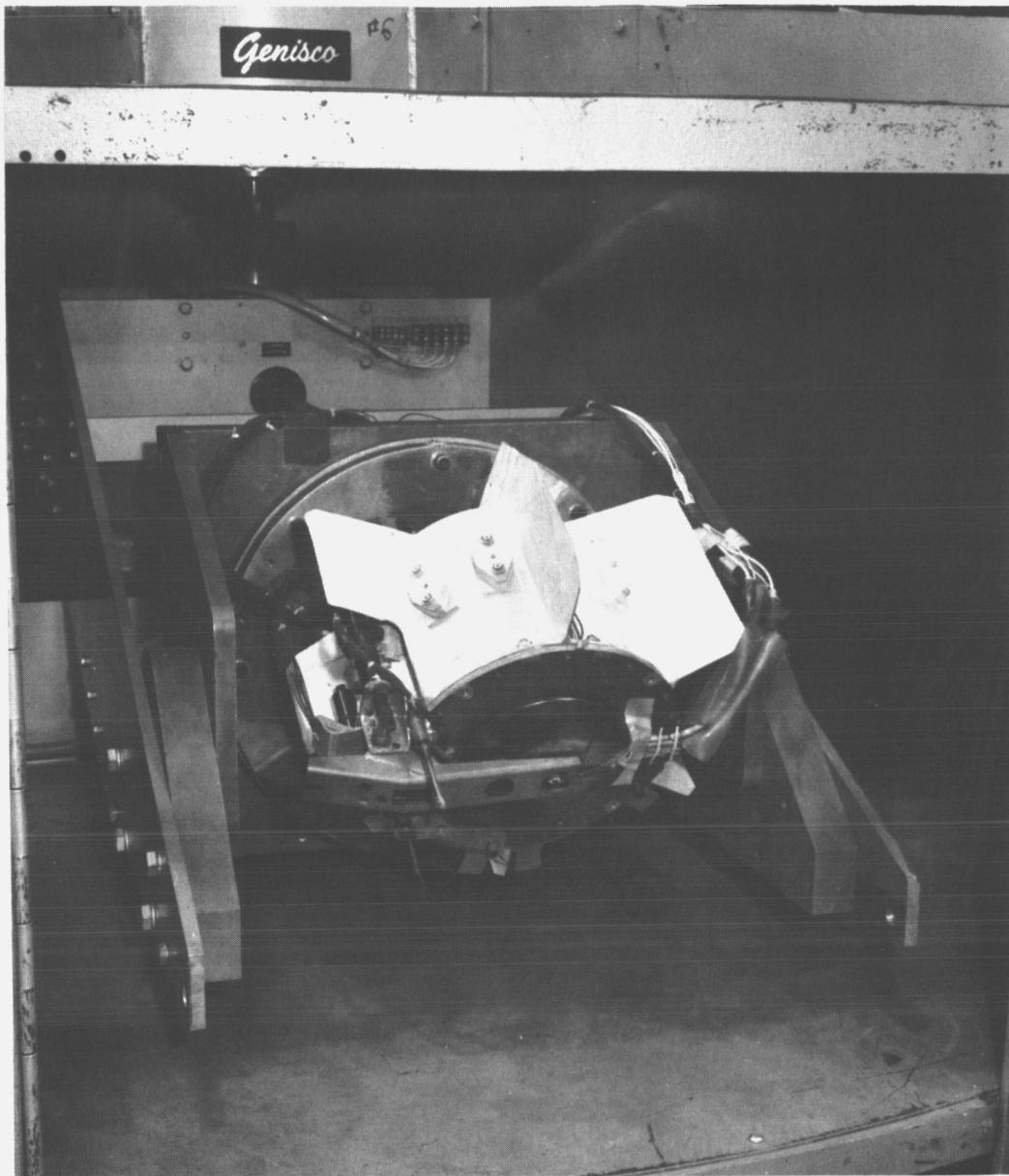


Fig. 13. S/N 1 Mockup Generator Acceleration Test

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After completion of Phase (1) and (2) testing, the generator was evaluated at lunar day thermal vacuum conditions in the S/N 1 configurations (Section B-2).

- (3) On completion of the thermal testing, the gold-coated foils were removed and the generator again dynamically tested. The vibration testing was performed at the qualification level with the door unlatched and allowed to float free. The generator was first vibrated in the vertical axis, then in an orthogonal axis.

For all tests, the specimen was mounted in the cast aluminum test fixture used in all previous SNAP 11 tests. This fixture was mounted directly to the head of the C-25HB shaker for vibration in the thrust axis, while the lateral axes of vibration were performed with the generator and fixture attached to the horizontal slide plate of the shaker. Shock tests were performed on a Hyge shock machine, and an auxiliary fixture was used to apply lateral shocks.

The thermal control system was fully operational, and was filled with NaK. The control system and generator were instrumented with thermocouples, installed upon assembly. Figure 13 shows the generator on the acceleration test apparatus.

c. Results

Electrical power for operation of the generator heaters was supplied by an a-c Variac. A wattmeter and voltmeter were provided for monitoring the input power.

Six thermocouples at points of special interest were selected for monitoring throughout the series of tests and were, (per Martin Dwg 431C1110003) as follows:

<u>T/C No.</u>	<u>Location</u>
2	Cold junction
1	Hot junction
5	Hot junction
7	Heater block
9	Heater block
38	NaK ring reservoir

During the first vibration test, hot junction thermocouple 1 apparently became detached, and gave erratic readings. Thermocouple 4, also

at the hot junction, was substituted for 1 during the remainder of the tests.

Heater power and voltage, as well as the six thermocouples, were read and recorded before and after each environmental exposure, and at intermediate intervals. In addition, the input current, input voltage, and temperatures were continuously recorded during each of the vibration and acceleration exposures by a recording oscillograph.

(1) Acceptance tests--locked shutter

Thrust axis vibration. During the three-thrust axis frequency sweeps, audible resonances were noted at approximately 70 and 250 cps. A lock nut at the bottom of one NaK cylinder had loosened during the first run, but was retightened. No failures occurred.

Lateral axis 1--vibration. A pronounced rattling noise, internal to the generator, was evident at 20 cps. After the third run in this axis, it was found that a lock nut was unscrewed from the shutter closing spring. This was replaced, and then did not again loosen.

Lateral axis 2--vibration. A strong shutter resonance occurred at about 40 cps, producing a displacement of approximately 1 inch double amplitude at the shutter door edge. A resonance at lower amplitude appeared at 100 cps. After the second run in this axis, the standard washer at the shutter arm locking bolt was replaced with one with a larger diameter to lessen the stress concentration in the arm at that point. No mechanical failures were observed.

Acceleration. The 6-g acceleration test was performed with no mechanical failures.

At the completion of the acceptance tests, operation of the NaK system was evaluated and the calibration was found to be acceptable. The shutter arm was again locked in the open position, and environmental testing resumed at qualification levels.

(2) Qualification tests--locked shutter

Thrust axis acceleration. Acceleration at the required qualification level was performed with no failures.

Thrust axis vibration. During a strong resonance at 60 to 70 cps in the first run, the shutter door exhibited a tendency to rotate about its center attachment bolt. This bolt was retightened, and a lock nut added. A lock nut was also added to the arm locking bolt.

After three vibration runs in the thrust axis, the shutter resonance appeared lower in frequency, the greatest response occurring from

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15 to 30 cps. The specimen was audibly noisy at about 100 cps, and what appeared to be smoke was visible near the point where the NaK tubes entered the generator casing. Upon inspection, this was found to be Min-K insulation. It was also noted on most of the ensuing runs.

The electrical output connector at the base of the generator loosened and dropped off during the fourth of the six runs in this axis, but was replaced and tightened with no permanent damage. Thermocouples 2, 4, 5 and 7 became very erratic, either intermittently shorting, or open. Couples 9 and 38 continued to operate normally.

Lateral axis 2--vibration. Oscillation of the shutter was very severe in a resonance at approximately 40 cps, and the shutter plate again rotated about its center bolt. The smoke or powder evident on previous runs was noticed on several runs in the lateral axis, but was not present during all runs.

After the third run in lateral axis 2, the large washer at the shutter arm locking bolt was found to be bent. It was replaced with two washers, and a third was installed as a spacer between the arm and the locking bracket. The bracket itself was slightly bent. The shutter center bolt was again tightened.

During the fourth run, shutter oscillations at 40 cps were more severe than previously. The refrasil cloth insulation in the shutter door began to shred, and the top of the door was deformed around the area where it contacted with the arm. The washers at the locking bolt were again bent, and as a consequence, the spacer was removed and the double washer replaced with the original single one.

The fifth run produced further shredding of the refrasil insulation, and severe shutter oscillations at about 35 and 70 cps. The shutter locking bracket was tightened after this run and it appeared to reduce the severity of the shutter resonances.

Lateral axis 1--vibration. Qualification level vibration in lateral axis 1 continued the general deterioration of the shutter door. Severe motion of the shutter occurred in the region of 70 cps due to torsional bending of the arm.

During the third run, the small clips securing the NaK tube to the frame of the shutter mechanism loosened and fell off. The shutter plate continued to rotate about its center bolt during all vibration runs, although the bolt was again tightened, and deformation of the plate continued.

Inspection of the arm and locking bracket after the fourth run revealed an apparent weakening. However, tightening of the bracket bolts reduced the amplitude of shutter motions in subsequent runs.

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Qualification level shock test--three axes. The specified shock test was applied in the thrust axis and both lateral axes, with no change in the generator condition, as revealed by inspection.

At the completion of the qualification tests, the generator was again calibrated to determine any adverse effects on the thermal control system. The calibration was found to be still acceptable, and dynamic testing was resumed, with the shutter in the unlocked position.

(3) Acceptance tests--unlocked shutter

Thrust axis acceleration. The acceleration test at 6 g was performed with no failures or change in the condition of the generator.

Lateral axis 1--vibration. The vibration test was begun with stabilized temperature conditions in the generator. The shutter had assumed a position of 1/2 inch open as measured from the outer edge (opposite the hinge), and this position remained approximately constant throughout the first run. A shutter resonance appeared at 50 cps, but produced only a small amplitude, and a lesser response occurred at 70 cps.

The shutter had closed automatically at the start of the second run, and remained closed during the run except for some lifting at the 50 cps resonance. The third run was begun when the shutter had begun an opening cycle. The shutter opened to a maximum of approximately 1-1/4 inches during the low frequency portion of the sweep, and had closed to 3/4 inch at the end of the run.

Lateral axis 2--vibration. Resonance of the shutter in lateral axis 2 built up over the range of 50 to 70 cps, then cut off sharply at 70 cps. All three runs in this axis were begun with the shutter open 1/2 inch. During the runs, the shutter opened from 1-1/2 to 1-3/4 inches, then gradually returned to a closed position. No failures were experienced.

Thrust axis vibration. The first thrust axis frequency sweep was begun with the shutter partially open. It had closed upon reaching approximately 100 cps.

The shutter was closed at the beginning of the second run, and due to the relatively low pressure in the NaK system, the arm and shutter plate assembly experienced "pumping" at the lower frequencies (10 to 50 cps).

For the third run, pressure in the NaK cylinders was high enough to open the shutter 3/4 inch at the start. With this restraining influence, no pumping occurred, and a shutter door resonance at 50 cps produced only a small amplitude.

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(4) Qualification tests--unlocked shutter

From the foregoing series of tests, it was evident that less damaging resonances were experienced by the shutter door and arm in the unlocked case.

Thrust axis vibration. Six thrust axis runs were made as specified, with the shutter unrestrained. In the instances when runs were started with the shutter closed, strong pumping was experienced at low frequencies. During runs started with the shutter open, the shutter experienced far less pumping, due to the damping action of the NaK fluid. Only slight damage to the shutter door was experienced.

The shutter appeared to operate properly throughout the first five runs, but at the conclusion of the sixth, operation was very stiff, probably due to distortion of the pivot-bearing alignment. Approximately 25 in. -lb of torque were required at the pivot to open the shutter and 11 in. -lb to close it.

Calibration of the NaK system was again performed.

(5) Additional qualification vibration--unlocked shutter

The thermal control system installed in generator S/N 1 was repaired and subjected to further vibration tests to prove the launch capabilities of the unlocked shutter configuration.

Six thrust axis vibration runs at qualification levels gave results similar to those obtained previously, although the shutter action was stiffer than its original operation.

The generator was reoriented and vibration begun in lateral axis 1. A strong resonant response at the shutter produced shutter door rotation and some shredding of the insulating material during the second run.

The third lateral vibration run produced a break in one section of the NaK system 62 seconds after the start, and at a frequency of 100 cps. The break occurred below the point where the tubing enters the top of the generator housing. Testing of the system was discontinued.

The shock and acceleration tests produced no failures and no detectable change in the NaK system. All degradation experienced by the test specimen was a result of vibration tests, primarily the combined sine and random qualification tests.

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Vibration tests performed with the locked shutter produced, in general, the greater part of the damage to the shutter door. Greater resonant response was produced by the mass of the shutter door when rigidly cantilevered in the locked configuration. Degradation consisted primarily of shredding of the shutter insulation, deformation and rotation of the shutter door, and possible deformation of the locking bracket.

While the unlocked shutter vibration tests provided a less severe environment for the shutter itself, the forces which did not build up in the shutter were transmitted to the NaK cylinders. The ultimate effect of these forces cannot be fully evaluated since a complete three-axis qualification test with unlocked shutter was not attainable. It appears possible that the unlocked condition causes a more severe load on the shutter pivot bearings.

The shutter tested exhibited the desired tendency to repeat a regular opening and closing cycle when operating with a constant heater power input. This cycle produced the differences in shutter action noted during the unlocked vibration tests. When a run was started at a higher NaK ring temperature, thus with the shutter in transit, the higher NaK pressure provided restraint and damping to limit shutter motion. In cases where the shutter was closed, the lower pressure permitted the shutter to respond more freely to vibration. The process of thermal cycling was speeded during the lower frequencies of vibration, since the relatively large shaker amplitudes introduced more air into the generator, with consequent rapid cooling.

The gold plating on the surfaces of the shutter door and heat shields withstood all tests with no visible damage.

At the completion of the flight acceptance level tests with the door locked open the generator calibration (determined as described in the Twelfth Quarterly Report) was found to be unchanged. Calibration was again checked and found to be unchanged at the end of the qualification level tests.

After completing both the flight acceptance and qualification level tests with the door unlocked and allowed to float free, the thermal control system was shown to be out of calibration. The shutter door had developed excessive friction in the shutter bracket bronze bushings. Disassembly of the shutter door mechanism revealed galling between the bronze bushings and the shutter door stainless steel actuating cams.

It should be brought out at this point that the loss of calibration, demonstrated after the unlocked series testing, is believed to be due to a thermal excursion during the calibration check rather than the dynamic testing. The actuator piston travel was inadvertently inhibited during the calibration check, resulting in a pressure buildup in the

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NaK system exceeding the pressure rating of the bellows in the piston assembly. For the degree of calibration loss (system yielding) noted, the shutter door would not have remained open during the vibration test, as observed. Therefore, it was concluded that system yielding occurred after dynamic tests.

In the final tests, vibration in the vertical axis at qualification levels with the shutter door free-floating proceeded without incident. However, during tests in the first orthogonal axis, the right side (right piston--upper NaK ring) of the thermal control system began to leak NaK at the welded joint which attaches the NaK ring to the tube from the actuator piston assembly. Inspection showed no galling or excessive friction in the actuating cam-bushing area. The NaK leak developed during the 69th vibration sweep performed and failure could have been caused by fatigue considering the more than two million cycles accumulated on the system during vibration testing. Figure 14, taken after all tests were completed, shows the shredded reffrasil cloth on the shutter door and the leaking NaK tube.

In summary, the existing design of the thermal control system appears adequate, at the required dynamic test levels, when the shutter door is latched in the fully open position.

However, when the shutter door is allowed free motion, shortcomings have been observed. On one occasion galling of the bronze shutter bracket bushing and attendant intolerable friction between the actuator cams and bushings were experienced. On another occasion, failure of a NaK tube was experienced. Therefore, the object of future dynamic testing on the S/N 4 modified generator will be to determine if the generator can be qualified with the door floating free, a desirable characteristic since it eliminates the need for a latching and release mechanism.

#### 4. Thermal Control System Calibration

Upon completion of the S/N 4 modified generator life tests, the generator was further reworked to improve the thermal coupling between the hot junction and NaK rings. Information gained from testing the S/N 1 generator mockup was used to make minor design changes to the upper portions of the generator. Therefore, this S/N 4 (modified)<sup>2</sup> generator was essentially the S/N 4 modified with a newly filled thermal control system and minor modification.

The generator thermal control systems were filled with NaK and evaluated at temperature in a thermal mockup prior to installation into a generator. Faulty filling operations can be detected and corrective action taken before the thermal control system is assembled into

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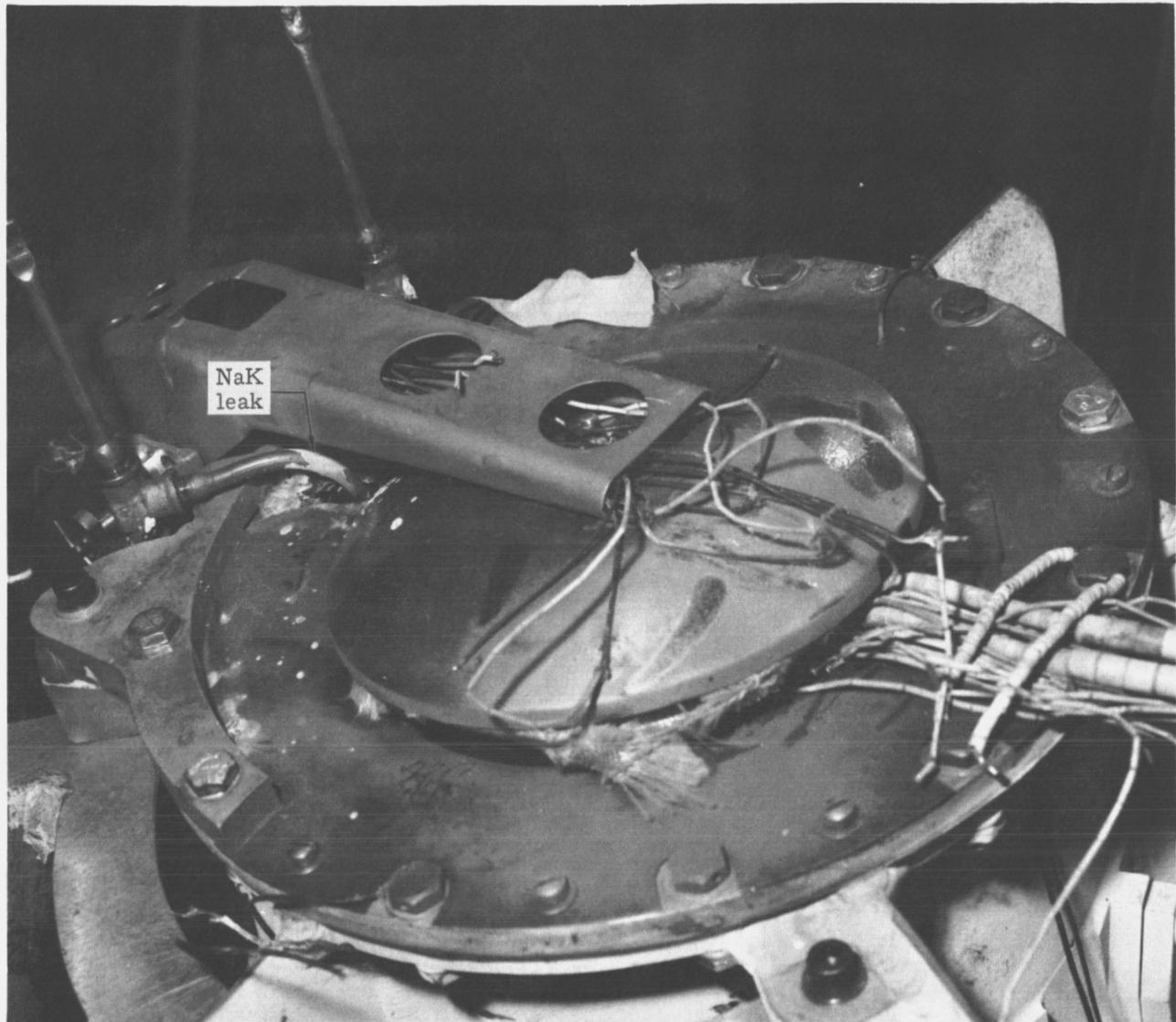


Fig. 14. S/N 1 Mockup Generator After Dynamic Tests

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the generator. However, a final adjustment under lunar night conditions is necessary to assure opening of the shutter door at the desired hot junction temperature.

The first major task was to adjust the thermal control system shutter to open at the proper average hot junction temperature. The generator was operated under lunar night conditions, in a vacuum tank, and the flexible bellows adjusted via mechanical penetrations and linkages. With the door completely closed an average hot junction temperature of approximately 925° F was established. The permanent flexible NaK system actuating bellows were then adjusted by the mechanical linkages so that the door just opened. The generator was then removed from the tank, the temporary adjusting linkages removed from the actuating pistons and a calibration point established in air. The success of the adjustment operations was demonstrated on several occasions for lunar night operation.

#### 5. S/N 4 (Modified)<sup>2</sup> Generator Flight Acceptance and Qualification Testing

The S/N 4 (modified)<sup>2</sup> generator is the S/N 4 modified generator with a replacement thermal control system and newly coated gold surfaces. These coatings were applied by using a fired-gold coating specified by Nuclear Process Specification NPS-03-037 consisting of stainless ceramic gold. Development of this coating is described in a later section of this report.

The objective of this test series was to subject a SNAP 11 generator of the latest design to acceptance and qualification level testing to verify the design, develop thermal control system calibration data and temperature distributions as a function of thermal input.

The S/N 4 (modified)<sup>2</sup> generator was first tested in the environmental high vacuum chamber. This chamber is cylindrical with overall dimensions of 30 inches in diameter by 30 inches in height. It is mounted in an upright position with a 24-inch diameter access door on the side. A Stokes 148C MicroVac vacuum pump with a rating of 37 cfm is used as a roughing and fore pump. The diffusion pump is an 8-inch V3C self-fractionating, three-stage pump with a capacity of 1000 liters/sec at  $10^{-4}$  torr. Both a water-cooled chevron baffle and a nitrogen-cooled baffle were used to reduce the migration of oil from the diffusion pump to the chamber.

The generator was suspended horizontally in a rack using three 1/32-inch stainless steel cables. The purpose of using a rack and cable arrangement was to minimize thermal losses from the generator support structures to the liquid nitrogen cold wall.

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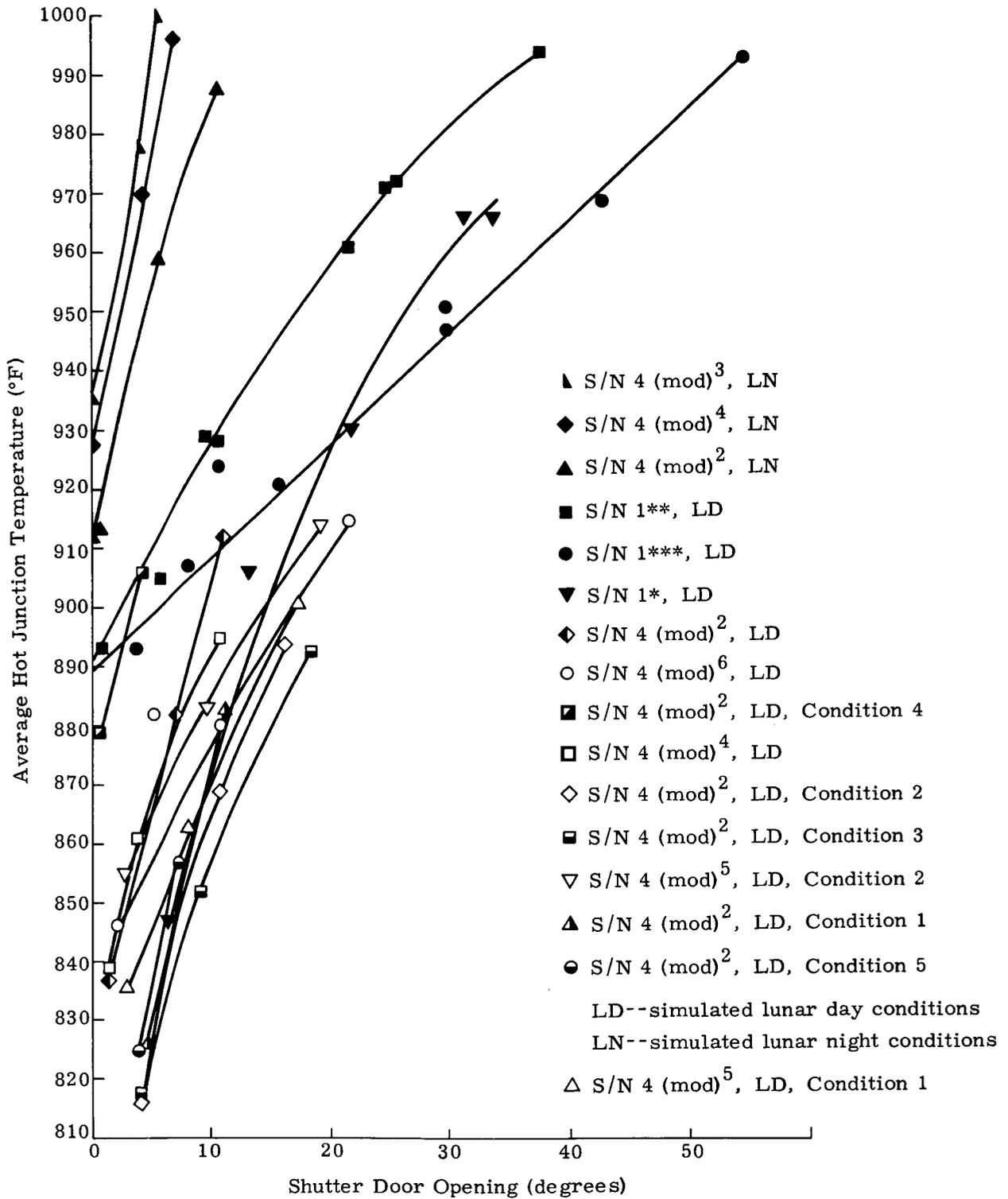


Fig. 15. Curves Comparing Thermal Response of Generators

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The initial operation was the adjustment of the shutter door to open at an average hot junction temperature of 925° F. The adjustment was performed using mechanical penetrations, as previously described, to adjust the NaK-filled bellows. The generator was then removed from the vacuum chamber to permit removal of the mechanical linkages and an air calibration checkpoint obtained.

a. Tests

The generator was then placed in the chamber and lunar night conditions of temperature and vacuum was re-established. The results for the S/N 4 (modified)<sup>2</sup> under lunar night conditions are shown in Fig. 15. For a limiting average hot junction temperature of 1000° F, the shutter door could be opened only about 11 degrees; the average hot junction temperature had increased from 912° F (0 degree door opening) to 988° F. The desired relationship between the hot junction and shutter door opening is as shown by S/N 1\*\*\*, the lunar day condition curve of Fig. 15 shows an increase in the hot junction of 83° F is required to open the door 45 degrees.

In an effort to improve the shutter door response, the insulating foils were moved from the annular cavity between the generator inner heat shield and inner can and reinstalled on the inside (inner periphery) of the inner shield, corresponding to the S/N 1 \*\*\*. The generator, designated S/N 4 (modified)<sup>3</sup>, was then calibrated at lunar night conditions so that the initial door opening was at an average hot junction temperature of approximately 925° F. Subsequent testing at lunar night, as shown in Fig. 15, revealed that the average hot junction temperature increased from 935° to 1000° F, and the door opened only 6 degrees.

The generator was again removed from the chamber and the lower end of the inner heat shield modified. This involved slotting the bottom of the skirt and bending half the tabs in toward the heater block. This allowed additional heat to be transferred, by radiation, to the NaK rings from the heater block. Results of the lunar night testing using this modification, designated S/N 4 (modified)<sup>4</sup>, showed that the hot junction temperature increased from 925° F (0 degree opening) to 995° F, on opening the door 8 degrees.

It should be brought out at this point that the thermal control system was operating mechanically as designed, i. e., for the change in the NaK reservoir temperature noted, the door was opening to the point required. The problem was in obtaining a sufficient increase in the NaK ring temperature to cause thermal expansion of the NaK to drive the actuating piston and thus open the door to dump excess power and control the hot junction temperature.

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After testing of the S/N 4 (modified)<sup>4</sup> at lunar night conditions, the generator was transferred to a second environmental chamber (used in the S/N 1 testing) to further investigate the thermal coupling between the NaK ring and hot junction under lunar day conditions. Before installation of the S/N 4 (modified)<sup>4</sup> generator in the tank, the NaK bellows adjustment screws were turned to the full-in position to cause a door opening at approximately 830° F to give additional test flexibility and to prevent a possible over-temperature of the cold junction. The results of testing at lunar day conditions are also shown in Fig. 15. The door opened at a lower hot junction temperature (approximately 830° F) as desired, but no improvement in the thermal coupling was evident. However, since the slopes of the curves were essentially identical, this indicated good correlation between lunar day and lunar night tests in two different vacuum facilities using the same generator.

The generator was then removed from the tank, the gold-coated foils were returned to the annular cavity between the inner heat shield and NaK rings, and the heat shield restored so that the generator was in the original S/N 4 (modified)<sup>2</sup> configuration. Subsequent testing at lunar day conditions showed no appreciable improvement in thermal coupling than that observed at lunar night. The S/N 4 (modified)<sup>2</sup> was then tested under five additional conditions in order to obtain additional thermal data:

Condition 1--The top external heat shield on the generator was further insulated by installing Min-K on top of the shield, thus reducing the heat loss from the NaK ring.

Condition 2--In addition to Condition 1, insulation was installed around the bolted flange which attached the outer shell and inner shell assemblies.

Condition 3--In addition to Conditions 1 and 2, insulation was positioned around the actuator pistons to reduce heat losses.

Condition 4--All the added insulation in Condition 3 was removed. In addition, the Triple A insulation between the NaK ring and top cover shield was removed, holes drilled in the original S/N 1 shield, and the shield installed.

Condition 5--The insulation between the NaK ring and cover shield was replaced and the original top cover shield re-installed. The thermoelectric converter section of the generator was then evacuated to simulate the condition of S/N 1\*\*\* during testing. Test results showed no improvement in coupling.

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Results of testing of these conditions are also shown in Fig. 15. Some improvement was noted upon adding the insulation, but no condition approached the coupling achieved in the S/N 1\*\*\* testing.

After the failure to obtain the desired coupling from these tests, a portion of the lower heat shield was removed to allow additional heat from the heat block to radiate directly to the NaK rings. This generator, designated S/N 4 (modified)<sup>5</sup> was tested without any insulating foils present (Condition 1). As evidenced from Fig. 15, some improvement in the thermal coupling was seen, but did not approach that desired. Installation of the foils (Condition 2) improved the shutter response slightly.

Since the NaK rings are not integrally connected to the inner can, the upper NaK ring was brazed to the can to improve heat transfer to the rings. (The bottom ring could not be welded because of the inaccessibility.) Testing of the configuration (designated S/N 4 (modified)<sup>6</sup>) did not show any improvement in shutter door response. Table 3 summarizes the changes made.

#### b. Conclusions

In summary, the testing performed late in this report period has not as yet approached the desired shutter response. However, an improvement has been made from the S/N 4 (modified)<sup>2</sup> lunar day condition which required a 75° F change in the average hot junction temperature to open the shutter door 10 degrees. The present S/N 4 (modified)<sup>6</sup> lunar day condition requires a 43° F temperature change. The change during the S/N 1\*\*\* was only 19° F.

The effort to resolve the coupling problem will be continued. Future plans call for testing with a copper conducting strap welded on the inner can between the NaK ring and top hot junction to reduce the thermal resistance between these two areas. Also planned is complete thermal isolation of the inner heat shield from the inner can to eliminate the negative temperature effect of the shield, i. e., decrease in temperature on the inner can as the shutter door opens.

### 6. Component Investigations and Development

#### a. Low emissivity gold coatings

To minimize thermal losses in the redesigned Q series generators, liberal use has been made of reflective heat shields. These shields and door parts require a low emissivity surface, 0.1 to 0.2, to be effective but unfortunately most metal oxides and other nonmetallic materials have emittances ranging from 0.5 to 0.9 at elevated temperatures.

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TABLE 3

Generator Configuration for Assigned Generator Designations

<u>Designation</u>	<u>Configuration</u>
S/N 1*	S/N 1 generator with newly filled TCS installed; gold surfaces electroplated (no intermediate ceramic coating).
S/N 1**	Same as S/N 1* except gold surfaces coated using newly developed process incorporating intermediate ceramic coating between gold and surface being coated. After S/N 1* testing, leaks in inner can resulted in vacuum in T/E converter section during vacuum testing.
S/N 1***	Same as S/N 1** generator except 14 layers of gold-coated foil were positioned around inner periphery of inner heat shield at plane of NaK rings.
S/N 4 (modified) <sup>2</sup>	S/N 4 modified generator with a newly filled thermal control system replacing inoperative one; newly coated gold surfaces used intermediate ceramic. Approximately 14 layers of gold foil installed around outer periphery of inner heat shield, in annular cavity between heat shield and NaK reservoir.
S/N 4 (modified) <sup>3</sup>	Same as S/N 4 (modified) <sup>2</sup> generator except foils moved from cavity to inner periphery of inner heat shield, i. e. , same position as in S/N 1***.
S/N 4 (modified) <sup>4</sup>	Same as S/N (modified) <sup>3</sup> generator except inner heat shield modified to allow additional heat to be transferred to NaK ring directly from heater block.
S/N 4 (modified) <sup>5</sup>	Same as S/N 4 (modified) <sup>4</sup> generator except for inner heat shield modification to allow more heat to be transferred by cutting off a portion of lower end of shield.
S/N 4 (modified) <sup>6</sup>	Same as S/N 4 (modified) <sup>5</sup> except upper NaK ring was brazed to inner can to improve heat transfer.

Therefore, effort was initiated to develop a low emittance coating for application to components of the SNAP 11 generator to minimize the radiant heat losses from the generator. The coating should be able to withstand, initially, exposure to air at 1000° F for extended periods and subsequent exposure to vacuum at 1000° F without change

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or degradation of the emissive coatings. The noble metals, i. e., gold, silver, and platinum will not form oxide films at elevated temperatures and will therefore retain the necessary low emissivity at elevated temperatures. It was therefore desirable to explore the application of gold coatings on a stainless steel base for the low emissivity requirement.

Development. A cursory survey of the literature on the emittance of various coating materials showed that oxides and other nonmetallic materials have emittances ranging from 0.5 to 0.9 at elevated temperatures whereas most metals and alloys give low emittance values at low temperatures or when measured in vacuum or inert atmospheres at elevated temperatures. The emittance values of metals increase considerably when the metals are preoxidized or measured in air at elevated temperatures, due to the formation of oxides on the surface of the metals. The noble metals do not form oxide films at elevated temperatures and will therefore retain a low emissivity at elevated temperatures. For this study, it was planned to explore the application of gold coatings on a stainless steel base for the low emissivity requirement.

Previous work in this area involved the application of gold coatings on stainless steel parts by electroplating from an acid gold solution. The electroplated coating, as deposited, had a matte gold surface and appeared to adhere well to the base metal. Oxidation testing of the gold-plated stainless steel parts was accomplished at 1000° F in air. After exposure for 18 hours, the gold-plated specimens were completely discolored and no longer retained the gold surface color. The discoloration was probably due to one or more of the following effects:

- (1) Contamination of the acid gold electroplated surface and oxidation of the contaminants,
- (2) Interdiffusion of the gold and stainless steel with subsequent oxidation of the diffused stainless steel at the surface,
- (3) Oxidation of the stainless steel through the permeable gold coating.

In all cases, the resulting oxidation products mask the gold color and increase the emittance of the coating.

The use of a ceramic coating applied directly to the stainless steel to act as a diffusion barrier between the gold coating and the stainless steel, and also to serve as an oxidation protective coating for the stainless steel, was investigated. The gold coating would be applied over the ceramic coating as a liquid bright gold solution, which on heat treatment will form a thin gold film. The gold film would then serve

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as the low emittance coating or as an electrically conducting layer to which the electroplated gold may then be applied.

Ceramic base coatings. The ceramic base coating is to serve as a diffusion barrier between the gold and stainless steel, and also as an oxidation protective coating for the stainless steel. The requirements for the ceramic base coating are as follows:

- (1) Good adherence to the stainless steel.
- (2) Compatible with the liquid bright gold solution.
- (3) Softening point of the coating to be  $> 1000^{\circ}$  F.
- (4) Protect the stainless steel from oxidation.
- (5) Have low vapor pressure under vacuum at  $1000^{\circ}$  F.

Three commercial ceramic coatings for stainless steel were investigated on this program. These coatings were:

- (1) Pemco 65-R-375, a black-base coat enamel which is fused to the stainless steel base at  $1480^{\circ}$  F.
- (2) Pemco 65-R-376, a white cover-coat enamel which is fused to the stainless steel at  $1450^{\circ}$  F.
- (3) Solaramic 5210-2C, a chromium oxide base, green enamel which is fused to the stainless steel at  $1750^{\circ}$  F. This coating was designed specifically for corrosion and oxidation protection of stainless steel components. No experience is reported with this coating in a high temperature vacuum environment.

Gold coating. The requirements for the gold coating are as follows:

- (1) Metallic coating must be formed below fusion temperature of the ceramic base coating.
- (2) The gold coating must be continuous and completely cover the base enamel coat.
- (3) Coating must be adherent and resist normal handling.
- (4) Coating must not be affected by heating in air at  $1000^{\circ}$  F or by heating in vacuum at  $1000^{\circ}$  F for extended periods.
- (5) Coating must have a low emissivity at  $1000^{\circ}$  F.

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For the present application, the liquid bright gold is painted on the enameled stainless steel as a very thin film heated slowly to 1000° F in air and held at that temperature for 30 minutes. The coated piece is then removed from the furnace and allowed to cool in air.

X-ray diffraction analyses of the heat treated liquid gold bright coatings on the ceramic substrate showed the coating to have a good, undistorted and unstrained gold phase. The X-ray diffraction analysis also showed that the gold phase was very uniformly distributed over the ceramic substrate in that no diffraction lines of the enamel substrate were recorded.

In addition to the liquid bright gold solution, a liquid bright platinum solution was also evaluated for this application.

Testing and observations. The evaluation of the ceramic enamel base coats and the liquid bright metal coatings were made by observing the condition of the coating after exposure to air at 1000° F and to vacuum at 1000° F for extended periods. Emissivity measurements were made on those samples which appeared promising after these tests.

Table 4 lists the coating materials and combinations of coatings tested during the course of this program.

The Pemco enamel R-376 produced a glossy enamel surface when fused on the stainless steel. The liquid bright gold coating when heat treated on the R-375 black enamel surface showed good coverage and adherence, and had a high gloss surface. Specimens of this type showed little change in appearance after 224 hours at 1000° F in air. The normal spectral emittance of the specimen measured at room temperature after 224 hours exposure at 1000° F was determined to be 0.03.

After exposure of the same specimen to a vacuum of  $10^{-6}$  mm at 1000° F for 108 hours, the ceramic enamel base coat was irregular and pitted, which was probably due to the boil-out of some of the low boiling constituents of the glossy coating. The gold coating also became dull and discolored from the vacuum treatment which was assumed to be caused by contamination of the gold with the low boiling materials in the glass. No emittance measurements were made on the vacuum-treated specimen due to the poor appearance and discoloration of the specimen.

Attempts were made to seal the enamel surfaces by the multiple application of thin gold coats. Specimens were prepared with five and 10 layers of gold coating on the Pemco black enamel. Both specimens had clean gold surfaces which were unaffected by an oxidation test of 64 hours at 1000° F. However, upon exposure to vacuum at 1000° F for 108 hours, the coatings on both specimens cracked and flaked off the stainless steel base.

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TABLE 4  
Summary of Testing Performed on Low Emissivity Coatings

Test No.	Base Coat <sup>(1)</sup>	Cover Coat <sup>(2)</sup>	Coats (No.)	Thermal Testing (hr)		Appearance After Thermal Testing
				Air (1000° F)	Vacuum (1000° F)	
1-1	White enamel	Gold	1	224	--	Gold coat clean with dull matte surface.
1-3	White enamel	Gold	2	224	--	Same--total emissivity at 1000° F = 0.04.
1-3a	White enamel	Gold	2	224	108	Gold coating blistered and cracked--no good.
2-1	Black enamel	Gold	1	224	--	Bright gold finish.
2-4	Black enamel	Gold	2	240	--	Same--total emissivity at 1000° F = 0.03.
2-4a	Black enamel	Gold	1	240	108	Gold surface dull, discolored & rough.
1-2	White enamel	Gold	1	16	--	Electroplate gold over bright gold coat. Coating discolored after thermal test, probably oxidation of contaminants.
2-3	Black enamel	Gold	1	--	--	Not able to electroplate gold over bright gold.
3-1	Green	Pt	1	64	--	Pt coat bright but flaking off.
3-2	Green	Gold	1	240	--	Excellent appearance, clean & bright gold coat.
3-2a	Green	Gold	1	240	108	Excellent appearance, clean & bright gold coat.
11	Black	Pt	1	224	--	Pt coating dull, streaky and discolored.
13	Black	Gold	1	16	--	(SS surface acid cleaned before coating.) Enamel coating blistered during testing due to HC1 boiloff.
14	White & black	Gold	1	224	--	Dark and wrinkled surface, probably interaction of two base coats.
15-1	None	Gold	1	224	--	Clean surface with scattered oxide spotting.
15-2	None	Pt	1	64	--	Pt coat discolored.
15-1a	None	Gold	1	224	108	Gold surface discolored over entire surface.
16	None	Gold	5	64	--	Clean gold surface with scattered oxide spotting.
16a	None	Gold	5	64	108	Coating badly discolored.
17-1	Preoxidized	Gold	1	--	108	No gold coating left, apparently diffused into base.
30-1	Black	Gold	5	64	--	Gold coat clean with slight film on surface.
30-2	Black	Gold	5	64	108	Entire coating (enamel & gold) flaked off due to thermal spalling.
31-1	Black	Gold	10	64	--	Same as 30-1.
31-2	Black	Gold	10	64	108	Same as 30-2.
G-1	Green	Gold	1	240	--	Gold coating appears clean and bright.
G-2	Green	Gold	1	240	108	Gold coating appears clean and unchanged. Total emissivity at 1000° F = 0.086.
17-2	None	Gold	1	--	108	No gold coating left, apparently diffused into SS base.
G-3	Green	Gold	2	--	108	Gold coating bright and clean.
G-4	Green	Gold	3	--	108	Gold coating bright and clean.
G-5	Green	Gold	3	--	108	Gold coating bright and clean.
G-6	Green	Gold	1 <sup>(3)</sup>	240	250	Gold coating slightly discolored. Total emissivity at 1000° F = 0.219.
G-7	Green	Gold	1 <sup>(4)</sup>	240	250	Gold coating clean and bright. Total emissivity at 1000° F = 0.086.
G-8	Green	Gold	1	240	--	White film formed on gold surface, used for chemical analysis.
G-9	Green	Gold	1	240	--	White film formed on gold surface, used for chemical analysis.
S-1	0.0001-in. pt	0.0005-in. gold <sup>(5)</sup>		168	168	Gold coating discolored.
S-2	0.0001-in. pt	0.001-in. gold <sup>(5)</sup>		168	168	Gold coating blistered.
S-3	0.0001-in. pt	0.002-in. gold <sup>(5)</sup>		168	168	Gold coating blistered.

(1) White coat--Pemco enamel 65-R-376 fused to SS at 1450° F.  
Black coat--Pemco enamel 65-R-375 fused to SS at 1480° F.  
Green coat--Solaramic 5210-2C fused to SS at 1750° F.

(2) Gold coat = Hanovia Liquid Bright Gold-Type MM bonded at 1000° F.  
Platinum coat = DuPont Liquid Bright Platinum bonded at 1000° F.

(3) Gold coating applied as 1 heavy coat.

(4) Gold coating applied as 1 very thin coating.

(5) Samples supplied by outside vendor.

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The Pemco white enamel R-376 also produced a glossy enamel coating when fused to the stainless steel. The liquid bright gold coating, when matured on this enamel, had a matte surface which may be due to some chemical incompatibility between the two coating materials. Extended heat treatment in air at 1000° F for 224 hours had no effect on the gold coating. The total normal emittance for this specimen at 1000° F is 0.04. Vacuum testing of this sample at 1000° F and  $10^{-6}$  mm for 108 hours resulted in severe blistering of the enamel undercoat and cracking and peeling of the gold coat.

The Solaramic green enamel, when applied as a coating to the stainless steel and fused at 1750° F, produced a less glossy surface than did the Pemco enamels. Depending upon the thickness of the enamel applied, it is possible to have a matte surface that ranges from 0.5 to 1 mil in thickness to a semigloss surface of two to four mils thickness. Consequently, the liquid bright gold coating will also vary in texture and gloss depending on the thickness and surface condition of the base coat. Oxidation testing of 0.001-inch thick stainless steel foil and 0.010-inch stainless steel plates coated with the Solaramic enamel and liquid bright gold coating for periods of up to 250 hours showed no effect on the gold surface. Subsequent vacuum testing of the same specimens for 108 and 240 hours, respectively, at 1000° F also produced little or no change in the appearance of the gold coatings. Normal spectral emittance of Specimen G-2 is shown in Fig. 16. The total normal emittance for the specimen was found to be 0.093 at room temperature (70° F) and 0.086 at 1000° F. The ceramic coatings on the two specimens described were approximately 0.0005 inch thick on the one-mil foil specimen and two to three mils on the 10-mil plate specimen. The gold coating on both specimens was very thin to achieve maximum adherence to the base coat. An additional sample was tested in which the ceramic coating was approximately two mils thick on a 10-mil stainless steel plate and a heavier gold coating was applied in a single application. The gold coating of this specimen after the air oxidation and vacuum tests at 1000° F for 240 hours and 250 hours, respectively, did not appear as clean and bright as the previous specimens. It is believed that the gold coating was somewhat less adherent when applied in a single heavier coating and, as a result, there was a portion of the gold coating lost during the vacuum testing. The specimen had a sufficient amount of gold coating left on the surface to warrant emissivity testing and the spectral emittance curve for this specimen is shown in Fig. 16. The total normal emittance of the coated sample was 0.236 at room temperature and 0.219 at 1000° F.

Upon removal of the gold coated Solaramic 5210 enameled specimens from the long term oxidation test, it was noted that a thin white film had formed over the gold surface. The film was adherent in that it was not readily removed by buffing but it could very easily be removed by water or rubbing with a dampened cloth, indicating the high solubility of the film in water. Analysis of the film by emission spectroscopy revealed that the following constituents were present:

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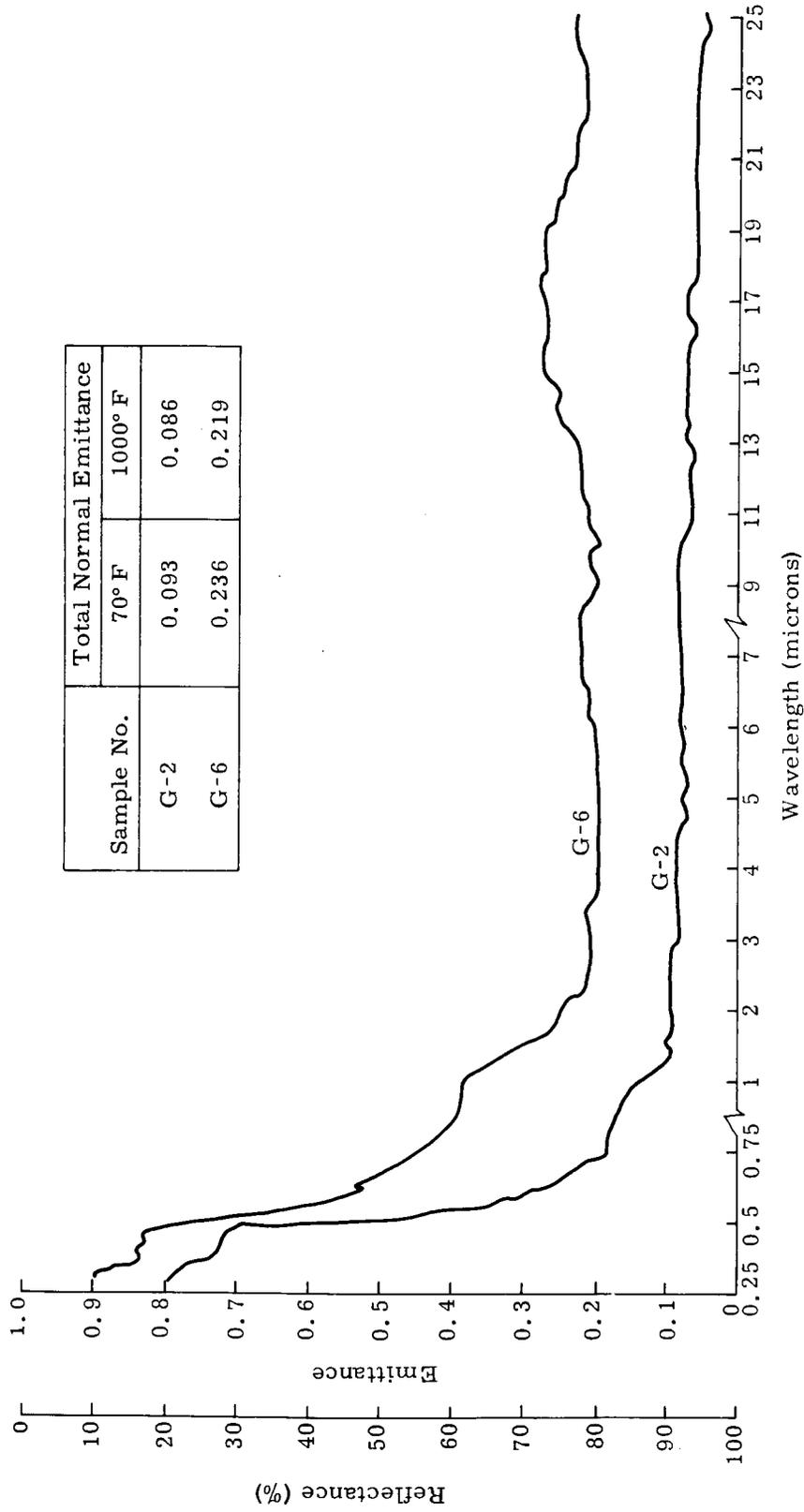


Fig. 16. Spectral Emittance of Gold on Solaramic Green Enamel

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Major components	Au (as expected from gold coating)
Minor components	Mo, Na
Subminor components	Fe, Si, Mg, Ca, Al, Ni, Cu
Trace components	Ti, Mn

The presence of sodium can probably be attributed to the enamel substrate as can most of the subminor constituents. The presence of molybdenum is attributed to volatility of the  $\text{MoSi}_2$  furnace heating elements in the furnace used for the heat treating of the enamel substrate, gold coating and for the oxidation testing of the coated specimens.

No emittance determinations were made on the specimens which had the film formation; however, it was noted that the film was completely removed by the vacuum treatment at  $1000^\circ \text{ F}$  and  $10^{-6}$  mm.

In addition to the evaluation of gold over the enameled substrate, it was also decided to evaluate the liquid gold bright solution coating directly on nickel and stainless steel panels. The coating was applied in the same manner as that used for the coating of the enameled panels and the same temperature cycle was used for the heat treating of the panel. The heat treating of the gold on the nickel panel for 1/2 hour at  $1000^\circ \text{ F}$  produced an oxidized surface with little indication of gold being present at the surface. This was probably due to the rapid diffusion of gold into the nickel substrate. The gold coating on the stainless steel panel appeared bright and clean after the initial heat treatment at  $1000^\circ \text{ F}$ . Continued oxidation testing at  $1000^\circ \text{ F}$  produced some scattered pin-point discoloration on the gold surface; however, after vacuum treatment at  $1000^\circ \text{ F}$  for 105 hours, the coated specimens had discolored completely and no signs of the gold coating were evident. A similar test was performed with a stainless steel panel which had been oxidized prior to applying the gold coating to determine if the oxidized coating would act as a diffusion barrier similar to the enamel. It was found, however, that the preoxidized coating was not effective as a diffusion barrier and the gold coating completely diffused into the base during the vacuum heat treatment.

In another series of tests, three samples were provided by a vendor. These contained a 0.0001-inch thick electroplated Pt base coat and a 0.0005-, 0.001-, and 0.002-inch thick electroplated gold coatings on each sample. The samples containing the 0.001- and 0.002-inch gold coatings blistered in the early stages of the oxidation tests and showed some slight discoloration of the gold coating. The blistering is probably due to expansion of occluded gases and/or salts from the electroplating process. The 0.0005-inch gold coated samples showed considerably less blistering but began to discolor, probably due to diffusion

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and oxidation. Vacuum testing of these samples had little effect on the blistering of the samples but did result in further discoloration of all specimens.

Conclusions. The use of a ceramic enamel coating on stainless steel has been effective in serving as a diffusion barrier between the gold coating and the stainless steel, and, in addition, the enamel coating has prevented oxidation of the stainless steel substrate. A Solar-amic 5210-2C enamel coating, fused at 1750° F, has withstood the oxidation and vacuum testing without deleterious effects. Lower temperature fusing enamels, while stable in air at 1000° F for extended periods, were not resistant to the vacuum testing at 1000° F and degraded considerably during the vacuum treatment.

The liquid bright gold solution coating applied over the ceramic enamel coating has successfully withstood 250 hours each of air oxidation and vacuum testing at 1000° F without degradation of the coating. A total normal emittance at 1000° F of 0.086 has been measured for oxidation and vacuum tested gold coating over a Solaramic enamel coated stainless steel substrate which exceeded the objective of 0.1 to 0.2 at 1000° F. A Nuclear Process Specification has been issued for this process and the process incorporated into the SNAP 11 program.

b. Mica evaluations

The applicability of mica as a thermoelectric hot shoe electrical insulator and the suitability of various micas has been examined in detail. This study was performed to determine the electrical resistivity as a function of temperature of six types of natural mica and the effect of heat aging. Also included was the chemical analysis of the conductive coating found on a sample of mica taken from the SNAP 11 S/N 4 generator.

The electrical resistivity of the following materials was investigated as a function of temperature:

- (1) Muscovite mica (Brazilian Ruby, India Madrea Green, India Ruby, standard Grade C as used in SNAP 11.
- (2) Phlogopite mica (dark and light).

The basic compositions of the micas tested are as follows:

<u>Type</u>	<u>SiO<sub>2</sub></u>	<u>Al<sub>2</sub>O<sub>3</sub></u>	<u>K<sub>2</sub>O</u>	<u>MgO</u>	<u>Fe<sub>2</sub>O<sub>3</sub></u>	<u>H<sub>2</sub>O</u>
Muscovite	45.2	38.4	11.8	--	--	4.6
Phlogopite	40.8	26.9	12.7	7.6	12.0	--

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The resistivity measurements made before and after heat aging utilized a simple, two-terminal, electrode configuration. The electrode consisted of aquadag circles, approximately 1.125 inches in diameter, painted on opposite sides of the mica. When the dielectric samples were ready for evaluation, they were sandwiched between the two contacting electrodes (stainless steel, one inch in diameter, with Hanovia type MM gold bright painted and fired onto the contacting surfaces). Voltage was then applied to the lower electrode (also instrumented with a chromel alumel thermocouple for measuring sample temperature) with a 14-volt battery. The current flow through the sample was measured by determining the voltage drop across a precision 5000-ohm, temperature-compensated resistor using a cubic digital voltmeter and amplifier with a gain of 100. Voltage was applied to the sample until no change in current flow was observed during a five-minute period.

The largest single source of error in determining the electrical resistivity as a function of temperature of any dielectric is the determination of the area-to-thickness ratio. An error in thickness ( $t$ ) of 0.0005 inch in this instance can change the ratio by approximately 25%. The effect on the log resistivity, however, is slightly more than 0.1 decade, noticeable, but not significant. The remainder of the measurements (voltage, current, temperature) are considerably less than the 25% figure quoted (voltage and current 1/2%, temperature 1%). The overall measuring accuracy is established at approximately 25% for this test.

At 1000° F, the lowest resistivity was exhibited by the Phlogopite dark mica,  $8.9 \times 10^8 \Omega\text{-cm}$ , as compared to  $2.5 \times 10^{10} \Omega\text{-cm}$  measured for the Phlogopite light, Muscovite, India Ruby and Muscovite Grade C. Figure 17 shows resistivity as a function of reciprocal absolute temperature after heat aging for 672 hours at 1016° F. The Phlogopite light mica showed little change in resistivity as a result of heat aging. The greatest change was exhibited by the Muscovite India green mica which decreased from  $1.75 \times 10^9$  to  $8 \times 10^7 \Omega\text{-cm}$ . In general, with the exception of the India green mica, the volume resistivity of the mica insulation tested ranged between  $3 \times 10^9$  and  $4 \times 10^{10} \Omega\text{-cm}$  at 1000° F before and after aging.

All samples of mica that were heat aged showed some evidence of delamination, a rainbow pattern and changes in thickness of as much as 0.0006 inch. The delamination was probably responsible for the changes in thickness.

Samples of mica from the original S/N 4 generator were examined for signs of deterioration and changes in electrical conductivity. The characteristic rainbow pattern indicated some delamination was ob-

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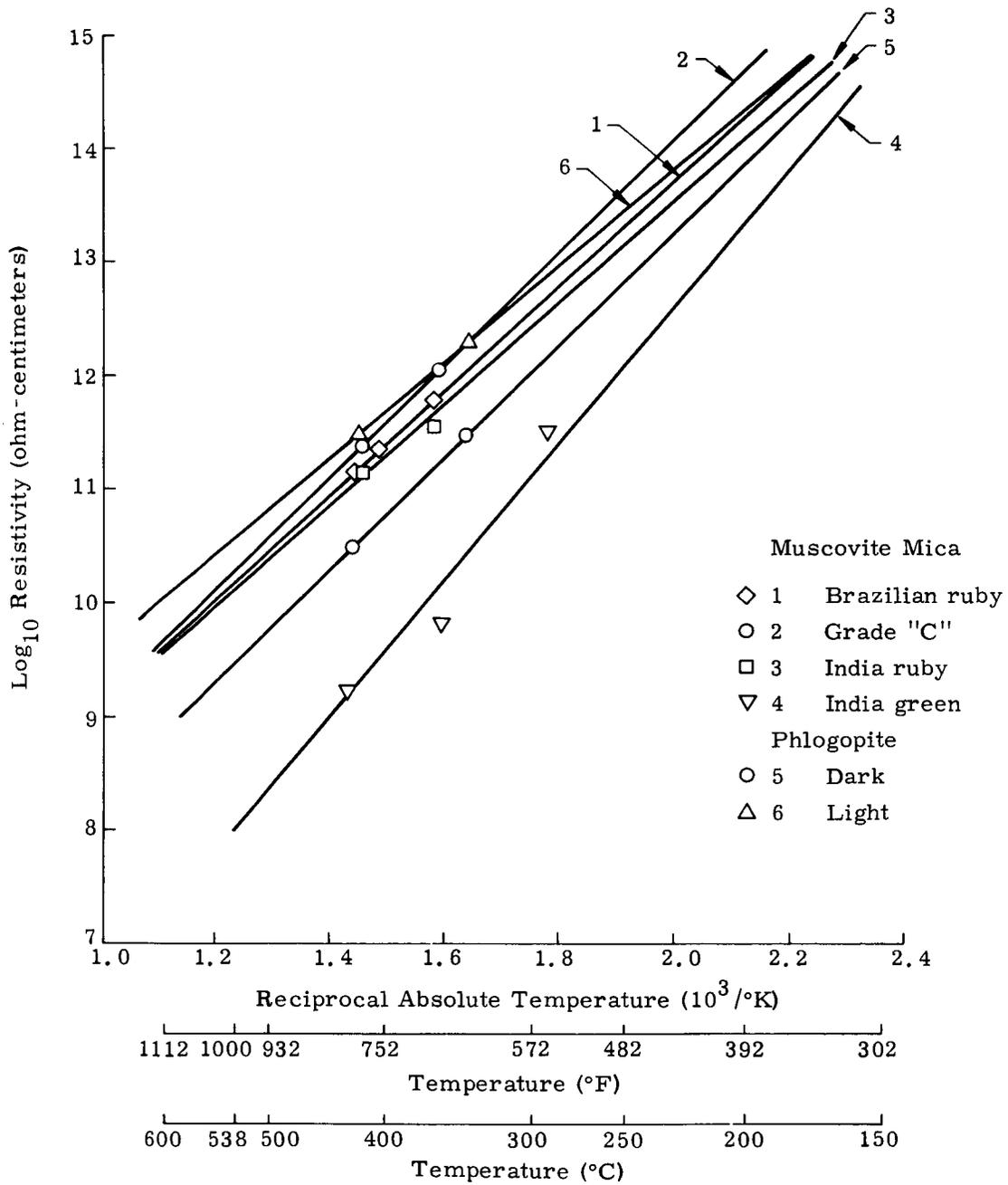


Fig. 17. Resistivity After Aging for 672 Hours at 1016° F in Air

served as well as a coating. The sample surface resistance between two aquadag strip electrodes was determined to be 180,000 ohms at room temperature. The elevated temperature volume resistivity was determined by the method previously described. The sample resistance at 374° F was found to be  $1 \times 10^6$  ohms and steadily increasing, indicating that the surface contaminant was either vaporizing or oxidizing from the surface of the mica. The resistivity as a function of temperature was found to be approximately 1/4 decade below that indicated for Grade C mica, in Fig. 17. The data for this test are given:

Temperature (° F)	Temperature ( $10^3 / ^\circ K$ )	Resistivity (ohm-cm)	Log Resistivity (ohm-cm)
701	1.55	$3.3 \times 10^{11}$	11.52
727	1.52	$2.3 \times 10^{11}$	11.36
792	1.44	$9.5 \times 10^{11}$	10.98

A sample of mica from the S/N 4 generator and a fresh sample of Grade C mica were chemically analyzed. The results and a comparison with an analysis of aluminum grease which has been used as a heat transfer media on the generator cold end hardware are shown in Table 5.

TABLE 5  
Elemental Composition Relative Concentration Level  
(gaseous elements excluded)

Sample	Major	Minor	High Trace
Coating on mica	Si, Al	Fe, Na, Ti, Sr	Ca, Ba, Mg, Mn, Nb, Pt, Rh
Grade C sample	Si, Al	Fe, Na, Ti, Mg, Ba	Va, Mo, Cu, Ni, Zr, Sr, V, Pb
Aluminum grease	Al	Fe, Na, Si, Sn, Cu, Ag, Pb	Ca, Cr, Mn, Ni, Ti

On the basis of the qualitative analysis, it is difficult to precisely determine the contaminant. However, both the used mica sample and the aluminum grease contained calcium. The Grade C sample which had never been subjected to generator environments contained some of the minor and high trace elements common to both aluminum grease and the contaminated sample, and therefore the analysis should be considered inconclusive. There appears to be no distinct advantage in

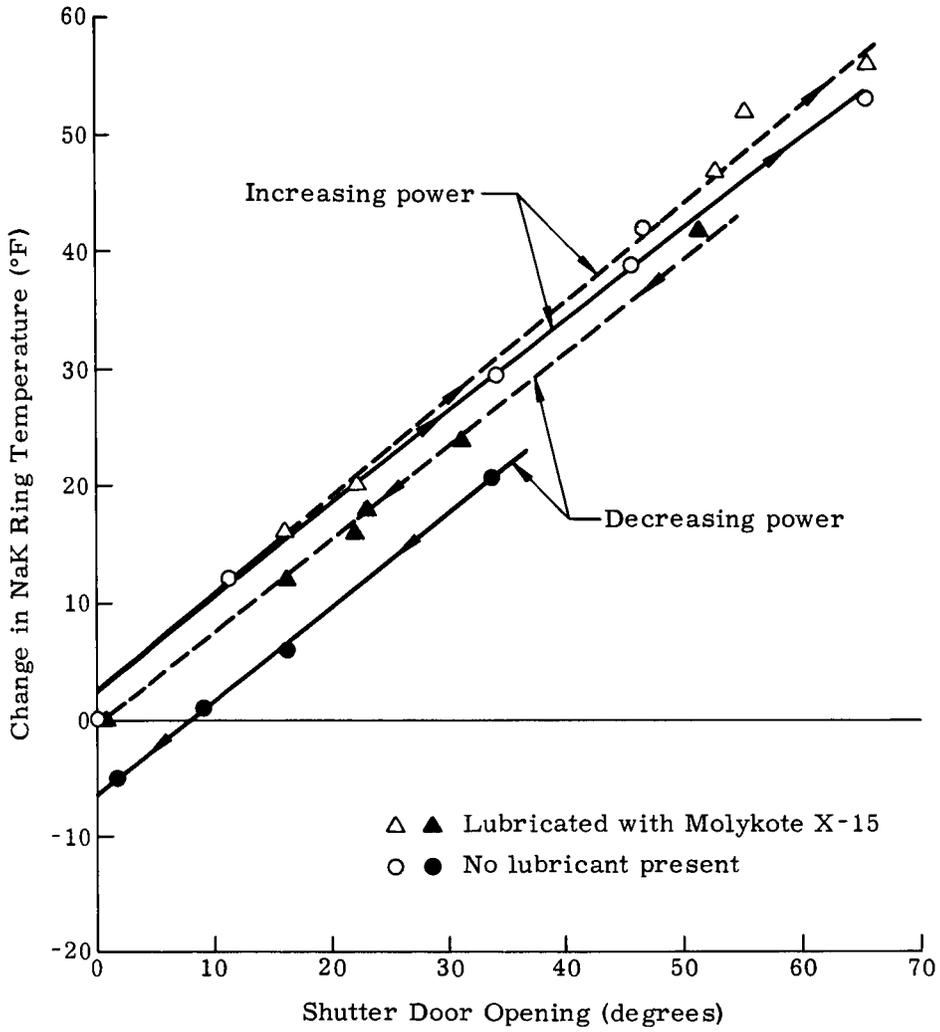


Fig. 18. Hysteresis Curves to Determine TCS Action With and Without a Lubricant on Connecting Links

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changing to another type of mica because the degradation of the Grade C mica is not felt to be significant at the operating temperatures presently used in the SNAP 11 generator.

c. Lubricant evaluation

Evaluations to determine the influence of Molykote X-15 lubricant on the operation of mechanical linkages in a thermal control system and effects on shutter door hysteresis were performed. The lubricant is rated for use under high vacuum and gamma radiation at temperatures ranging from  $-300^{\circ}$  to  $1200^{\circ}$  F. In the test program, the performance of a shutter door without any type of lubricant and with Molykote X-15 was compared.

To test the shutter door action without any type of lubricant, all of the shutter door linkage was ultrasonically cleaned in a caustic solution to remove surface film and any traces of lubricant present. The test data obtained, shown in Fig. 18, indicate that a NaK ring temperature approximately  $10^{\circ}$  F lower than that required to initiate door opening is necessary to close the door.

To determine the effect of adding Molykote X-15 lubricant, the connecting links were ultrasonically cleaned in a caustic solution, washed using a dilute solution of Radosol 501 detergent in water, water rinsed and dried in a vacuum oven. Subsequently, Molykote X-15 was brushed on the linkage parts and allowed to dry in air for the minimum recommended 24-hour period.

Testing showed that the  $10^{\circ}$  F hysteresis noted in testing without lubrication was reduced to  $3^{\circ}$  F (Fig. 18).

Based on the test results, Molykote X-15 will be applied to the connecting links between the actuator pistons and shutter door cams. This requirement has been added to the design drawings and any subsequent change in linkage lubrication will be incorporated by DCN to the design drawing.

d. Thermal control system NaK loading

During this report period, a third thermal control system was successfully filled and evaluated. This operation has now become routine and repeatable. Three successive systems have been successfully loaded with NaK with repeatable results.

After the TCS filling, system hardness and temperature response were determined as for previously filled systems. The degree of system hardness was determined by imposing various vertical loads (at constant NaK temperature) on the actuator pistons and measuring

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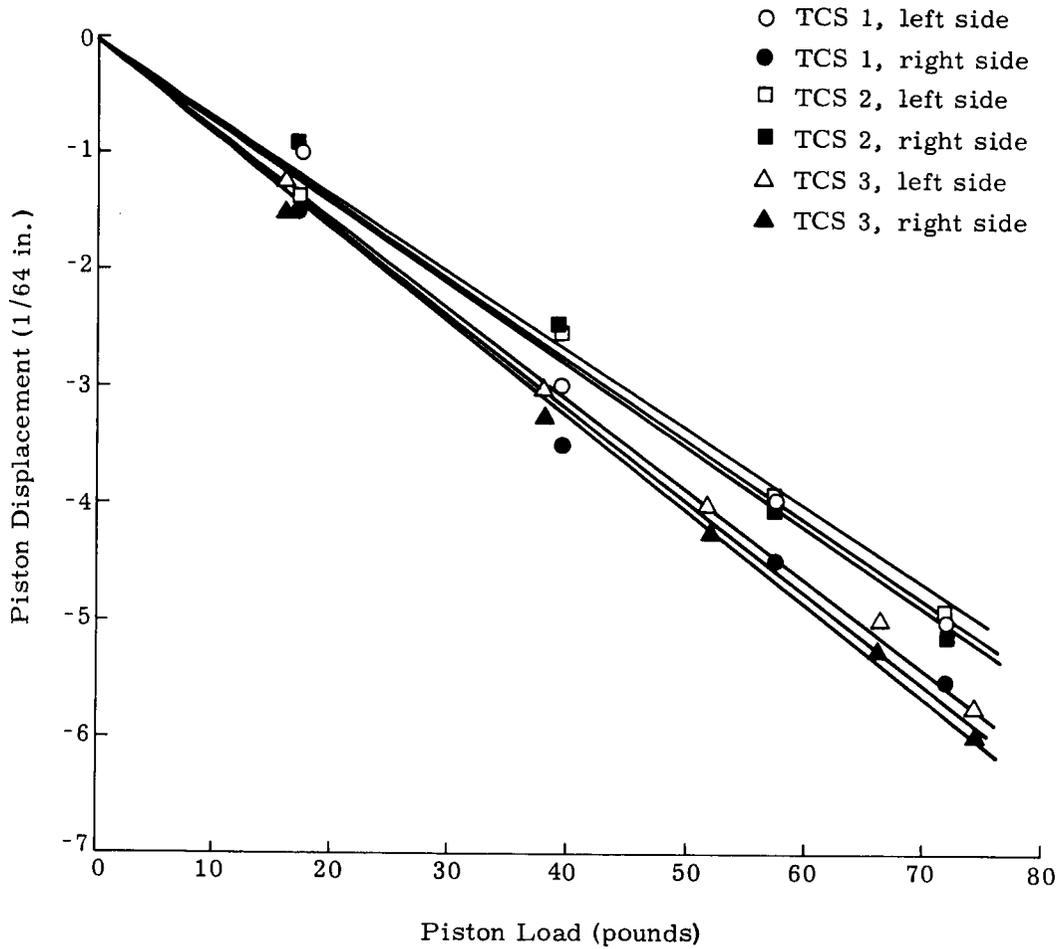


Fig. 19. Piston Displacement Versus Piston Load Curves for TCS 1, 2, and 3

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the resulting piston displacement from the no-load position. To determine system temperature response, a vertical load of 64 pounds (approximately that load required for one piston to activate the shutter door independently) was imposed on each piston and the piston displacement as a function of the NaK ring temperature determined. These are considered to be the two major characteristics with which to evaluate each TCS and allow comparison of individual systems.

The data obtained from the filling of TCS 3 are compared with the two previously evaluated TCS (TCS 1 and 2) in Figs. 19 and 20. From the figures it can be seen that the systems filled to date are almost identical. The piston deflection for a 70-pound load for all six individual NaK systems is seen to be approximately 5/64 inch and indicates that the change in the NaK ring temperature required to fully open a TCS shutter door (70 angular degrees, 0.250-inch piston travel) is approximately 57° F.

e. Generator impurity analysis

The purpose of this analysis was to gain additional insight into possible sources of generator contamination and causes of power degradation. The analysis included gas chromatography of argon used in generator testing, zirconium used as a getter material removed from the S/N 4 generator, generator cold end hardware, gold coated copper electrical straps and cold trap liquid residues collected during initial outgassing of a generator.

Argon used in generator test operations was analyzed for oxygen and nitrogen content. For the most part, both oxygen and nitrogen were just detectable but not measurable quantitatively. The composite peak of total gases based on quantitative comparison with air as the standard gave an average of 0.4% air for four samples taken.

Three cold trap residues were analyzed when it was suspected that contaminants other than water were present. The first abnormal residue contained copper particles. These were traced to a copper line fitting job exterior to the device under test. The other two abnormal residues contained small irregular red particles. These particles were identified as small pieces of rubber from vacuum hose connections, all exterior to, and not affecting the device under test.

The zirconium getter materials removed from the S/N 4 prototype generator were subjected to elemental and gas analyses. No significant differences were found between the getters and during elemental analysis. Aside from the principal component, Zr, each contained: Fe 0.05, Al 0.10, Si 0.01, Cr 0.01, Cu 0.01, Mn 0.01, V 0.01, Pb 0.01, Mg 0.01, Ti 0.01.

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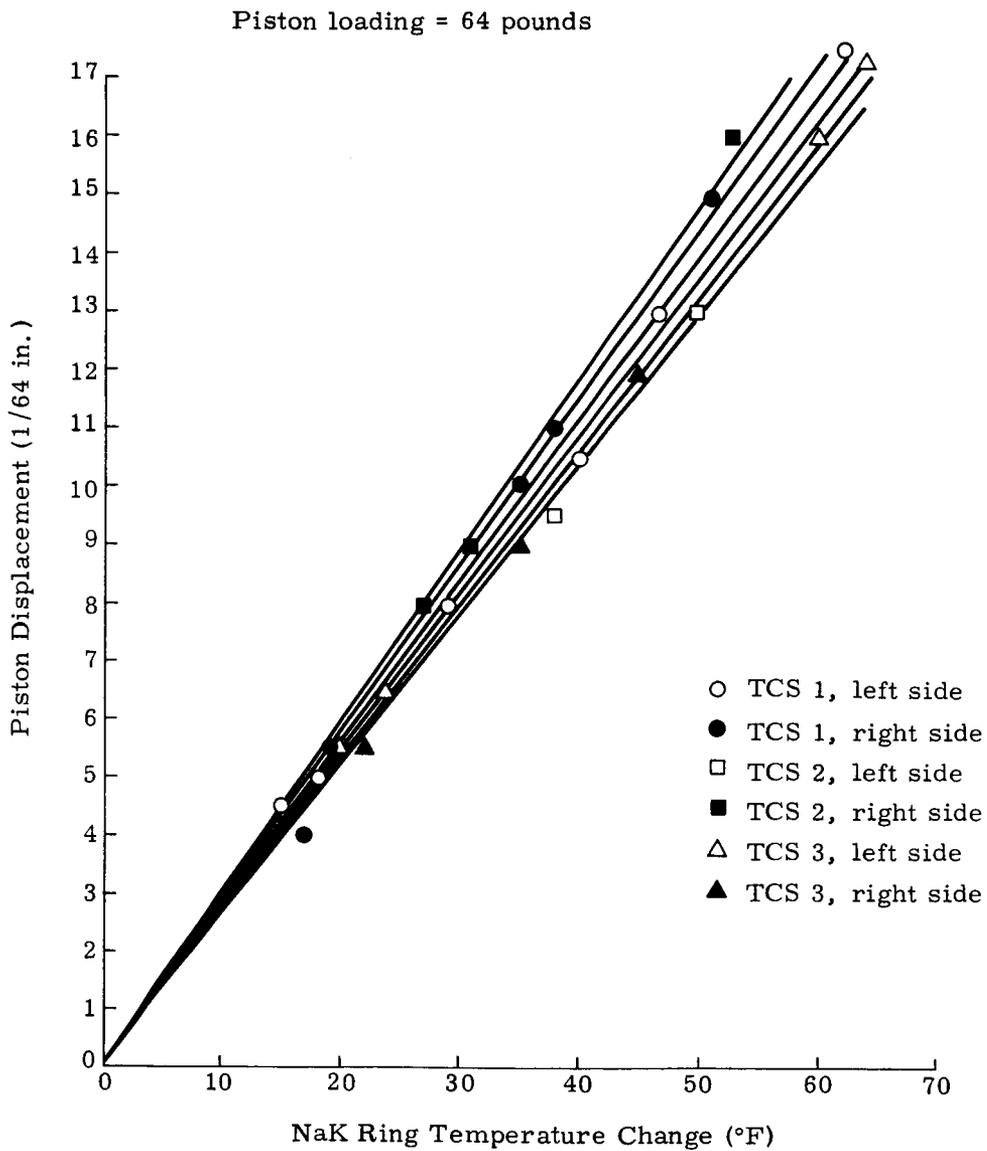


Fig. 20. Piston Displacement Versus NaK Ring Temperature Change Curves for TCS 1, 2, and 3

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Hydrogen analyses of the zirconium getter revealed a content of 0.65 wt % hydrogen for two individual samples.

A thermogravimetric analysis showed a very slight weight increase over the temperature range of 70° to 1100° F. The analysis was performed in argon and the very slight weight increase is attributed to pickup of O<sub>2</sub> from the argon atmosphere. It is significant that there was no weight loss indicating that there is no contamination volatilizing from the getter at generator operating temperatures. Oxygen analyses of two samples after 2300 hours generator operation were 3.80 and 4.49 wt % O<sub>2</sub>.

A carbon analysis revealed a carbon content of approximately 1475 ppm.

An aluminum heat sink bar, shown in Fig. 21, and used on the cold end of the thermoelectric module, was analyzed for elemental impurities and integrity of the anodized coating. The bar was cross sectioned and the coating thickness was measured on both the inner and outer surfaces. Anodized coating thickness averaged 0.002 inch. Abrasion tests of the material support the fact that the coating was hard and adherent. No weight change was detected on a sample of the anodized coating over the temperature range of 70° to 1100° F by thermogravimetric analysis. A spectrographic analysis of the coating for elements other than aluminum gave: Mg 1.0, Si 0.60, Zn 0.08, Cu 0.05, Cr 0.03, Mn 0.03, Zr 0.03, V 0.03, Fe 0.40, Ti 0.02, B 0.02, Mo 0.01, Pb 0.01 and Sn 0.01. Deposits found in all the cold end hardware cylinders were subjected to an elemental analysis which showed Al, Ca, Cu, Cd, Zn, Fe, Mg and Si to be present.

The gold-plated oxygen-free copper straps, attached to the cold ends of the thermoelectric elements and used as power leads, were chemically analyzed. Analysis for elements other than copper and gold revealed Fe and Co at low concentrations. A thermogravimetric analysis showed no weight loss up to 1100° F. After remaining at 1100° F for 1/2 hour, the gold coating disintegrated (without weight loss).

#### 7. Q Series Qualification Generators

Results of the development tests performed during this past quarter have led to design changes of the Q/N generators. These improvements are considered minor and are intended to improve generator performance, both thermally and electrically, without major modification of existing components fabricated earlier. A brief description of the generator design changes follows:

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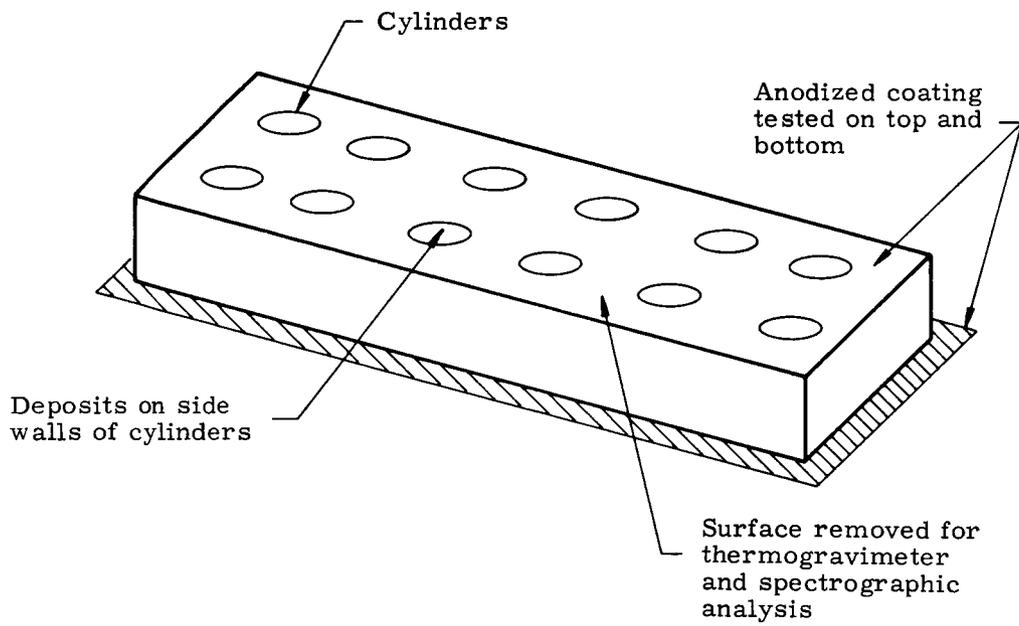


Fig. 21. Generator Cold-End Hardware

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- (1) All external, low emissivity, heat transfer surfaces will be coated with a ceramic gold coating developed and described by NPS-03-037, Fired Gold Coating. The need of a durable low emissivity coating for improved reflective heat shields to minimize heat losses and provide desired temperature distributions made this a desirable change.
- (2) The use of a fuel block with a 19/32-inch greater height than the electrically heated block required relocation of the heat baffle on the heat shield.
- (3) Distortion of the thermal control support assembly was evidenced during recent qualification level vibration tests, thus requiring strengthening of the bracket. Two gussets have been added to reinforce this assembly.
- (4) Reliability data on crimped electrical connections is more extensive than silver soldered connections and, in addition, are more structurally sound than solder at SNAP 11 operating temperatures. Therefore, silver soldered intermodule connections have been replaced with a mechanically crimped joint using a modified and gold plated Nicopress sleeve. This was done after bench testing of the crimp integrity and joint electrical resistance.
- (5) The 0.002-inch thick dimpled reflective foil insulation has been replaced with an improved gold coated reflective foil. The reflectors consist of 0.001-inch stainless steel foil with 0.006-inch dimples evenly spaced and dimpled foil coated with gold per the Fired Gold Coating, NPS-03-037.
- (6) To further reduce hysteresis in SNAP 11 shutter door motion, the chrome plated 17-4PH CRES shutter shaft bushings were replaced with uncoated SAE 660 bronze bushings. This has reduced friction and hysteresis effects. The shaft control slotted links were lubricated with Molykote X-15 for smoother operation of the control assembly.
- (7) To reduce thermal losses through the shutter door, use was made of alumina shutter door shoulder bushings. These have now been replaced with titanium to lessen the probability of breaking during dynamic testing.
- (8) Dynamic testing of the S/N 1 generator mockup with an operating thermal control system revealed several generator details which could be improved. Changes to the thermal control assembly were:

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- (a) A cotter pin was added through the shutter door bracket to eliminate rotation of the door caused by vibration.
  - (b) A jam nut was added to the shutter door attaching bolt and lock nut as a precaution against the lock nut coming loose during launching.
  - (c) A bolt, washer and lock nut were added through the NaK tube clamp and bracket. These replaced the original drive screw which made the assembly and welding of the NaK tubing difficult to disassemble after fitting.
  - (d) The purchased clamps for the NaK tubes were replaced with manufactured clamps to achieve a closer fit.
- (9) Life testing of the S/N 4 modified generator showed a gradual increase in internal resistance and resultant decrease in power output during the 127-day test. It is suspected that impurities contained within the internal components of the generator may be adversely affecting the thermoelectrics. Water vapor and oxygen in the thermal insulation, absorbed from the atmosphere during generator final assembly, are suspect. Consequently, the fabrication processes and assembly procedures of the Q series generators are currently being modified to reduce or eliminate potential sources of contamination. The major changes include:
- (a) Bakeout of the Min-K insulation in air at 1050° F to remove organic binders and water in lieu of the 930° F used in the past. This temperature is higher than the operating temperature of the generator. After bakeout in air, the insulation is transferred to a vacuum fixture and reheated to 1050° F, evacuated to 50 microns and backfilled with cold trapped argon at temperature. The insulation, along with other components, are transferred to an inert dry box and generator assembly performed as a dry box (oxygen-free) operation.

The Q/N 1 generator thermoelectric modules were fabricated earlier in the program using the 930° F bakeout temperature for insulation and then assembled in air. However, the inner can assembly, with attached modules, was heated to an intermediate temperature and evacuated prior to dry box final assembly. The Q/N 2 generator will be fabricated by out-gassing all insulations, including modules prior to the bonding operations. Module bonding and generator final assembly will use inert gas-dry box procedures.

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- (b) Aluminum grease, used as a heat transfer medium for conduction of heat between the module cold shoe heat sink and the outer can, will be eliminated.
  - (c) The cold shoe piston springs were replaced with a larger diameter spring wire to increase the pressure to a nominal 150 psi.
  - (d) The internal module assembly instrumentation was re-located to decrease the length of the electrical leads, and hence lead power losses, and eliminate the excessive notching of the Min-K insulation located in the top dome.
  - (e) Notes added to engineering drawing for more stringent reliability and quality control.
- (10) To remove the contact resistance of the electrical outlet male-female Cannon plug, the female end of the electrical connector has been removed, the external power leads soldered to the terminals and the exposed end of the male electrical connector potted with RTV-501.

Fabrication of details and subassemblies of the Q/N 1 and Q/N 2 generator, suspended for the past six months, was resumed in early April upon completion of the S/N 4 modified generator life test and the S/N 1 mockup generator thermal vacuum and dynamic tests. Essentially, all details of the Q/N 1 generator, including the thermal control system and outer housing, are complete. Final assembly of the generator was initiated and completion is anticipated by late May 1965. The Q/N 2 generator fabrication is scheduled to be complete by late June. Fabrication of Q/N 3, the final generator in this series, will be held in abeyance until Fiscal 1966 to take advantage of the Q/N 1 and Q/N 2 flight acceptance test results.

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### III. GENERATOR FUELING PROGRAM\*

#### A. OBJECTIVES

Oak Ridge National Laboratory (ORNL) has developed a curium-242 fuel compound for use in the SNAP 11 generator. A fuel loading of 900 watts(t) is to be available during August 1965. The AEC plans to utilize this fuel loading as a generator demonstration test. The test will be performed at ORNL on the Q/N 1 generator. Current test plans include a 120-day test in vacuum at simulated lunar temperatures. Martin activities to support the fueling demonstration and test were initiated late in this report period. The objectives for this past quarter were to:

- (1) Prepare an interface specification defining ORNL-AEC-Martin responsibilities in the joint fueling and test program.
- (2) Release design drawings, initiate procurement of materials and fabrication of fuel blocks.
- (3) Finalize fuel block assembly procedures.
- (4) Initiate design of fueling support equipment.
- (5) Initiate preparation of a safety hazards report in support of the ORNL fueling.

#### B. SCOPE

##### 1. Interface Specification

Specification MN-10135, "Interface Specification for Nuclear Fueling and Functional Testing of a SNAP 11 Thermoelectric Generator," has been prepared. In summary, the document defines areas of responsibilities in accomplishing encapsulation of the radioisotope fuel, assembly of the encapsulated fuel into the fuel block, installation of the fuel block into the generator and monitoring the electrical output of the generator after fueling.

##### 2. Fuel Block Design and Fabrication

The fuel block drawings were completed and released for procurement of materials. Specification MN-10121, "Fuel Block Inspection Criteria," covering assembly procedures and inspections during fueling,

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was prepared. This specification and copies of the fuel block drawings were forwarded to ORNL.

Bids on materials and fabricated subassemblies for the fuel blocks were received in late April. Procurement orders for all materials were placed and delivery will permit shipment of the assembled fuel block to ORNL in July.

In general, the SNAP 11 fuel block is a cylindrically shaped multi-material unit which occupies the internal volume of the generator. The TZM (molybdenum alloy) fuel capsule, fueled with curium-242 ( $\text{Cm}_2\text{O}_3$  in an iridium matrix) is located in the center of the fuel block. The capsule is surrounded by a platinum sphere, approximately 2-1/4 inches in diameter, which provides shielding and acts as an energy absorber for impact considerations. This assembly is enclosed in graphite and beryllium subassemblies to provide the proper thermal distribution and ablative protection.

### 3. Safety Analysis Hazards Report

Preparation of a safety analysis for inclusion in the ORNL Hazards Report was initiated in late April. The outline for this report follows:

- I. Introduction
  - A. Mission
  - B. Safety Philosophy
  - C. Safety Criteria
  - D. Summary
- II. Description of SNAP 11 Radioisotope Power Supply
  - A. Generator Description
  - B. Generator Performance
  - C. Converter Description
- III. Description of the Fuel
  - A. General Description
  - B. Cm-242 Fuel Form Description

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- C. Chemical Compatibility
- D. Toxicology
- E. External Dose Rates
- IV. Description of the Fuel Containment Structure and Fuel Loading Procedure
  - A. Capsule Geometry
  - B. Material
  - C. Fuel Loading and Positioning
  - D. Method of Closure
  - E. Generator Assembly Procedure
  - F. Physical Properties of Capsule Material
  - G. Pressure Buildup
  - H. Capsule Oxidation
- V. Generator Demonstration (testing)
  - A. Facility (other than hot cell)
  - B. Test Setup
  - C. Test Procedure
  - D. Generator Operational Test
  - E. Fueled Generator Maintenance
- VI. Nuclear Hazards Analysis
  - A. Thermal Considerations
  - B. Loss of Environment
  - C. Stress Evaluation
  - D. Impact Tests
  - E. Safety Mechanisms and Fail-Safe Devices

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#### IV. RELIABILITY ENGINEERING\*

##### A. OBJECTIVES

The reliability objectives for this period have been to achieve the closing of as many established problems resulting from the development test programs as possible and maintain reliability support in accordance with Reliability Program Plan, MND-2952-13.

##### B. SCOPE

###### 1. Task 1--Reliability Program Plan

Accomplishments of the past quarter are reported by task number in the following material.

###### a. Task 2--Design Specification

No effort in this area during this period.

###### b. Task 3--Apportionment and Prediction

The S/N 4 modified generator completed 3000 hours life testing during this period. The data from this testing were subjected to the same normalizing technique as that used in earlier studies. The results show that the generator had a power degradation or drift rate of approximately 3 to 4% per 1000 hours. As mentioned in the earlier sections of this quarterly report, certain design changes are being incorporated into the Q series generator design which should improve this situation. Spring pressures are being increased, selective fitting of pistons and cold sink bores, vacuum bakeout of insulation and dry box assembly will be accomplished.

###### c. Task 4--Failure Mode and Effect Analysis

Progress has been made in increasing assurance of acceptable leak rates of the inert gas contained in the thermoelectric compartment. A revised manufacturing process specification has been prepared which provides more specific details on the accomplishment of leak rate measurement techniques.

###### d. Task 5--Parts and Material Selection and Control

No effort this period.

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e. Task 6--Design Review

No effort this period.

f. Task 7--Trouble Reporting and Corrective Action

Fifteen problems of a reliability nature have been identified since September 1964. Thirteen of these problems have been analyzed and corrective action taken to close them. Those remaining open include P-element hot junction bond deterioration and determination of acceptable generator leak rates.

g. Task 8--Qualification

The engineering test procedure for the S/N 4 modified generator thermal vacuum and dynamic test program was reviewed and changes recommended.

h. Task 9--Vendor Surveillance and Control

No effort was expended this period since materials have been received for the Q series generators.

i. Task 10--Manufacturing Process Plans and Specification Review

The NaK filling and calibration procedure for the thermal control system was reviewed and approved for its present intended use. However, this procedure was established for limited quantities and would require simplification if it is to be applied against production quantities of generators.

j. Task 11--Preparation of a Demonstration Plan

Task completed.

k. Task 12--Reliability Training and Coordination

No special effort this period.

l. Task 13--Reporting

Internal documentation of the reliability effort was generated and recorded.

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